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Approach to Level 2 Analysis Based on Level 1 Results, MEG Categories and Compounds, and Decision Criteria

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16. ABSTRACT The report describes an approach to the decision criteria needed to proceed from the initial emission screening analysis (Level 1) to the detailed emission characterization (Level 2), and a Level 2 analytical approach. The decision criteria, considering only the available Level 1 chemical data, provide a basis which can be used for proceeding to a Level 2 emission characterization based on chemical correlation with compounds identified as Multimedia Environmental Goals (MEGs). The report discusses the types of Level 1 environmental assessment samples, and the chemical data available which can be prioritized for a MEG-based Level 2 plan. It presents a logic network for determining the need for a Level 2 sampling effort. It also presents an integrated approach to Level 2 inorganic compound analysis, an identification scheme consisting of characterization of the initial sample, of bulk composition, and of individual particles. Detailed logic networks are included to provide direction to the analyst during the identification process. The analysis of solid and liquid samples for organic compounds is discussed, using mainly combined gas chromatography/mass spectrometry. A logic network is provided for the organic analyst.

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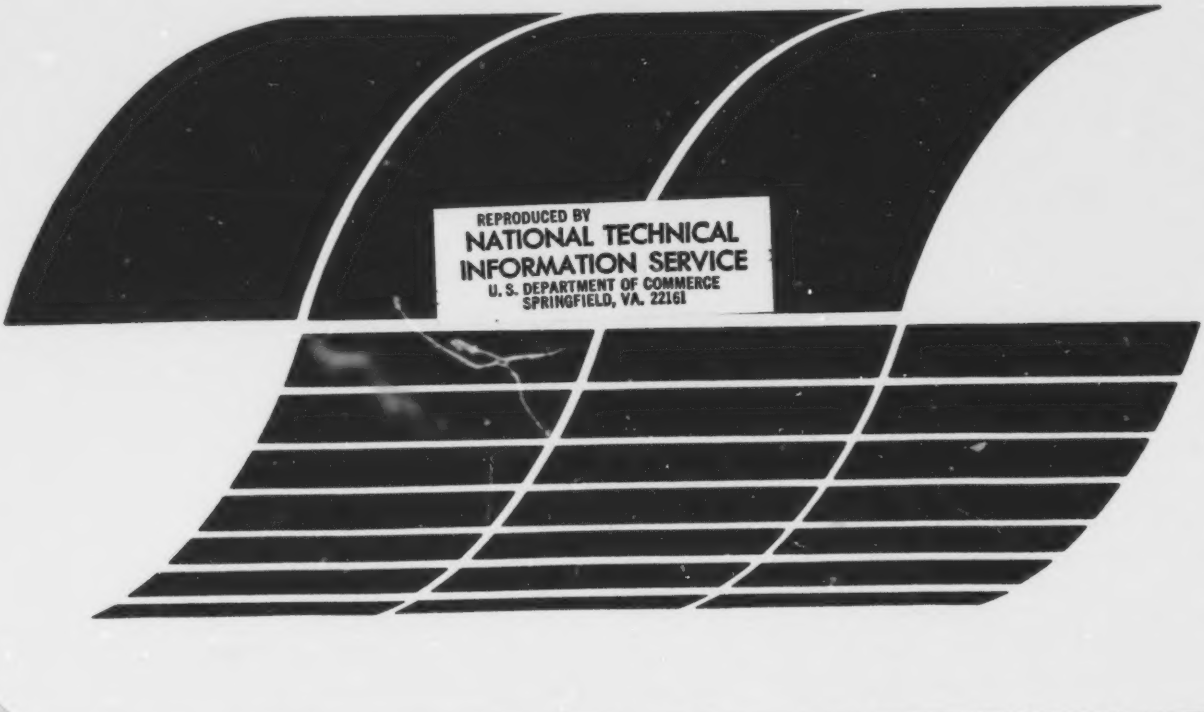

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Approach to Level 2 Analysis Based on Level 1 Results, MEG Categories and Compounds, and Decision Criteria

by

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SUMMARY

This report has been prepared as a requirement of Contract 68-02-2613, Task 6 to document a proposed approach for the interpretation of environmental assessment data and identification of additional analytical requirements. Volume I describes the general approach to Level 2 analysis and Volume II, a companion to this document, is the application of the approach to a set of samples. Addressed are the decision criteria needed to proceed from the initial emission screening analysis phase (Level 1) to the detailed emission characterization phase (Level 2) and the Level 2 analytical approach. This stepwise phased approach to environmental assessment has been shown to be significantly cost effective compared to a direct approach of obtaining the needed information by sampling all streams and analyzing them for all suspected pollutants (1).

In the phased approach to environmental assessment, Level 1 is the initial survey method by which hazardous streams are distinguished from those less hazardous or innocuous. Level 1 sampling and analysis have been designed to generate semiquantitative (±3X) information about the organic and inorganic elements of interest in the sampled stream so that a dominant offender does not go undetected (2). This information can then be used to prioritize the detailed and specific analysis required in Level 2.

The decision criteria developed in this report provide a basis which can be used for proceeding to a Level 2 emission characterization. However, this study has been reduced somewhat in scope by limiting the number of specific pollutants of interest to a chemical correlation with those identified as the Multimedia Environmental Goals (MEG) (3) compounds as defined by the EPA and detailed by Research Triangle Institute. The decision criteria presented here consider only the available Level 1 chemical data. The contributions from the bioassay results to the choice of Level 2 samples is not part of this data evaluation. The possibility of negative chemical results (that in which individual components do not exceed a toxic concentration) occurring with positive biological toxicity is highly probable. Therefore, consideration by the analysts of the bioassay results is an important part of a Level 1 environmental assessment and could result in a

Level 2 effort to detect synergistic agents. In recent environmental assessments (4) the correlation between chemical data and bioassay tests has been excellent though the entire biological test matrix must be conducted.

Section 2 of this document discusses the types of field samples, retainable samples, and chemical data avialable from a Level 1 Evironmental Assessment which can be prioritized for the MEG based Level 2 plan. Retained Level 1 samples are collected from each of the Source Assessment Sampling System (SASS) components, either in the form of solids, water samples, or methylene chloride washings. The samples collected are analyzed either in the neat or subsequently prepared form. These retained Level 1 samples are addressed as valid Level 2 samples. Any MEG organic categories or inorganic elements requiring a Level 2 sampling are discussed and some preliminary sampling recommendations made. Complete resampling, therefore, becomes unnecessary under this Level 2 approach, providing the uncertainty in the Level 1 sampling is tolerable for the required environmental assessment. A feature of the Level 1 analysis is that the data can be identified by the analyst when resampling is necessary. This is discussed in Section 4 where a logic flow is presented for establishing the need for a Level 2 sampling effort.

The sampling procedure for Level 1 Environmental Assessment recommends use of the Source Assessment Sampling System (SASS). This system consists of the following components:

- 1, 3, and 10 micron particulate cyclones
- Filter
- XAD-2 sorbent trap resin
- XAD-2 sorbent module condensate
- Impingers
- Probe and connecting lines

In the case of a low amount of collected particulate matter all solids may have been used during Level 1 activities. Therefore, Level 2 analyses cannot be performed on those neat samples. However, it is possible to proceed into this Level 2 approach with Level 1 retained prepared samples.

Section 3 presents the transitional details which start with Level 1 data and arrive at the decision to conduct Level 2 analyses. In this decision format Level 2 is only required at the specific Level 1 reporting points containing $\mu g/m^3$ ($\mu g/\ell$ or $\mu g/g$) concentrations which exceed Minimum Acute Toxicity Effluent (MATE) guidelines. The MEG compounds, decision criteria MATE, Level 1 reporting points, and generalized Level 2 tests are presented as usable Level 1 — Level 2 data reduction tables (Appendix A). Level 1 reporting points are given in Table 2-5 and specifically detailed in Table A-1. The decision to conduct a Level 2 analysis is then made based on Level 1 data and MATE concentrations within a specific MEG category.

Section 4 presents decision criteria for Level 2 sampling. If a Level 2 sampling effort is necessary, the SASS may not be the most viable train. This section, therefore, contains some preliminary suggested Level 2 sampling methods.

In Section 5 an integrated approach to Level 2 inorganic compound analysis, which is also being developed under Contract 68-02-2165, is presented. This identification scheme consists of:

- Initial sample characterization, where elemental composition, sample stability, and bulk morphological structure are determined.
- Bulk composition characterization, where qualitative and quantitative anion, oxidation state, and X-ray diffraction information are derived.
- Individual particle characterization, where single particle elemental composition, X-ray diffraction pattern and morphology are measured.

Detailed logic networks are also included to provide direction to the analyst during the identification process. The analysis of solid and liquid samples for organic compounds is discussed in Section 6. Combined gas chromatography and mass spectrometry is the main technique used to identify organic compounds in this plan. Direction is provided to the analyst by means of flow charts and written explanation on such items as:

- Sample size
- Mass ranges to be scanned

- GC columns to be used
- Estimated GC conditions for complete separation

Under EPA Contract 68-02-2150 a procedures manaual (5) is being prepared for Level 2 organic sampling and analysis. It should be referred to by the Level 2 analyst as a more complete compendium on organic compound identification methodologies.

The Level 2 approach presented here has been used to access Level 1 data reported by Battelle Columbus Laboratory from run #2 of a 6" Fluidized Bed Combustor Unit, EPA Contract No. 68-02-1409, Task No. 33. Level 2 analyses of these retained Level 1 samples has been conducted and reported (6) under EPA Contract No. 68-01-3152, Tasks 2, 3 and 4. The report from this analytical effort should be referred to for examples of specific method applications.

CONTENTS

Summary. iii
Figures. ix
Tables x
Acknowledgments. xi

1. INTRODUCTION 1

2. LEVEL 1 SAMPLES, NATURE OF RETAINED SAMPLES AND LEVEL 1 DATA 3
    2.1 Level 1 Samples 3
        2.1.1 Level 1 Home-Site Samples. 5
    2.2 Nature of Retained Level 1 Samples. 10
    2.3 Level 1 Data on Home-Site Samples 13

3. MEG CATEGORIES AND COMPOUNDS AT MATE CONCENTRATION AS DECISION CRITERIA. 19
    3.1 MEG Categories, Compounds and Elements. 19
    3.2 MATE Concentrations 20
    3.3 Level 1 - Level 2 Data Reduction and Decision Charts. 20

4. SUGGESTED APPROACHES FOR LEVEL 2 SAMPLING AND ON-SITE ANALYSES 22
    4.1 Decision Criteria for Level 2 Sampling. 22
    4.2 Suggested Level 2 On-Site Tests for Problematic MEG Compounds 24

5. LEVEL 2 INORGANIC ANALYSES 28
    5.1 Inorganic Compound Identification 28
        5.2.1 Initial Sample Characterization. 29
        5.2.2 Bulk Composition Characterization. 46
        5.2.3 Individual Particle Characterization 53

6. LEVEL 2 ANALYSIS OF RETAINED SASS SAMPLES FOR ORGANIC COMPOUNDS. 56
    6.1 Hardware Requirements and Options for Level 2 Analysis. 57
        6.1.1 GC/MS. 57
        6.1.2 Chemical Ionization (CI) Mass Spectrometry 58

CONTENTS (Continued)

- 6.1.3 Multiple Ion Detection Mass Spectrometry. 59
- 6.1.4 High Resolution Mass Spectrometry (HRMS). 60
- 6.1.5 Infrared Spectroscopy 61
- 6.1.6 High Pressure Liquid Chromatography (High Resolution Liquid Chromatography, HPLC) 61
- 6.1.7 Capillary GC/MS 61

6.2 Sample Preparation and Extraction Procedures 62

- 6.2.1 Probe Wash, Cyclones, and Filter SASS Train Samples 62
- 6.2.2 XAD-2 Sorbent Trap. 62
- 6.2.3 Extraction of the Condensate. 64

6.3 Analysis of the Extracts for Volatile Components . . 64

- 6.3.1 GC/MS Analysis of the Probe Wash, Cyclones, and Filter Extract. 64
- 6.3.2 GC/MS Analysis of the XAD-2 Module Extract 65
- 6.3.3 GC/MS Analysis of the Condensate Extract 66

6.4 Liquid Chromatographic (LC) Separation 68

6.5 GC/MS Analysis of the LC Fractions 70

- 6.5.1 Fraction A (1). 70
- 6.5.2 Fraction B (2 and 3). 71
- 6.5.3 GC/MS Analysis Fraction C (4 and 5) . . . 72
- 6.5.4 GC/MS Analysis of Fraction D (6 and 7). . 72
- 6.5.5 GC/MS Analysis of Fraction E (8). 75

6.6 Level 2 Analysis of Water Samples. 76

- 6.6.1 Direct Aqueous Injection GC/MS. 76
- 6.6.2 Purge and Trap Concentration Technique 78
- 6.6.3 Extraction of Water Sample for GC/MS Analysis. 79

Appendices

- A Level 1 Data Reduction and Decision Charts. 80
- B Liquid Chromatography Separation Procedure. 108
- C MEG Organics/Major Mass Peaks 111

References. 151

FIGURES

Number		Page
2-1	SASS Train Schematic.	11
2-2	Decision Logic for Phased Level 1-Level 2 Analysis.	15
4-1	Decision Logic for Level 2 Sampling	23
5-1	Initial Sample Characterization	31
5-2	Bulk Composition Characterization	33
5-3	Individual Particle Characterization.	35
5-4	Logic Flow Chart for Initial Sample Characterization. . . .	36
5-5	Logic Flow for Bulk Composition Characterization.	37
5-6	Logic Flow for Individual Particle Characterization	38
5-7	Level 2 Liquid Sample Compound Analysis Scheme.	39
6-1	General Logic Flow Chart for Level 2 Organic SASS Component Analysis.	63
6-2	Logic Flow Chart for Level 2 Organic Aqueous Samples. . . .	77

TABLES

Number		Page
2-1	Level 1 Analytical Categories.	4
2-2	MEG Compounds Not Contained in Level 1 Samples	6
2-3	MATE Level 2 Triggering Values for C1-C6	12
2-4	General Level 1 Reporting Points	16
4-1	Reactive Organic and Inorganic Compounds Capable of Being Identified by Specific Test Kits (8)	26
5-1	Summary of Recommended Procedures for Anion Analysis	42
5-2	Useful IR Bands.	47
5-3	Listing of Assigned Infrared Bands Observed in Particulate Samples.	48
5-4	Infrared Bands of Some Common Nitrates (cm^{-1}).	50
5-5	Infrared Bands of Some Common Sulfates (cm^{-1}).	51
6-1	LC Fraction Blending	69
6-2	Solvents Used in Liquid Chromatographic Separations.	69

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1. INTRODUCTION

This document has been prepared as a requirement of Task 6, Preparation of a Draft and Final Report on a Level 1/Level 2 Analysis Scheme, of EPA Contract 68-02-2613, Non-Personal Quick Reaction Engineering and Technical Services. The approach presents a detailed, but preliminary, Environmental Assessment Plan for characterization of the emissions from an energy process based on Multimedia Environmental Goal (MEG) organic and inorganic chemical species. The MEG compounds are potentially hazardous compounds which can be emitted from a variety of energy conversion processes under certain process conditions at concentration levels which could cause an environmental insult. The emissions characterized by compound class and elemental screening methods (Level 1) are used to identify those stream samples which may offer specific pollutant emission problems. The Minimum Acute Toxicity Effluent (MATE) values are used as the concentration decision criteria for determining whether additional detailed emission characterization (Level 2) should be performed. The Level 2 characterization is considerably more accurate and quantifies specific MEG inorganic and organic compounds. The Level 2 analysis plan presented here is considered to be preliminary because the information used to test and evaluate the decision criteria was not based on a recommended Level 1 analysis scheme (e.g., field test data were not obtained and provisions for stabilizing the low molecular weight species were not utilized). The value of the work reported here is (1) the generation of a practical reporting format, (2) the logic path network to make decisions for proceeding to Level 2 analysis, and (3) the initial approach at multimedia Level 2 analysis. Retained Level 1 samples have been the focal point of the Level 2 approach developed. Level 2 on-site analysis schemes are recommended for future programs to study the complexity of on-site volatile and reactive species identification. However, some approaches to Level 2 on-site compound identification are included as suggestions to the performing analyst.

The Level 2 analysis of retained Level 1 samples is complex. Decisions requiring analytical expertise follow every Level 2 data generation effort. Therefore, after each Level 2 analysis step the information obtained must be evaluated and the list of MEG compounds identified, quantified, tabulated, and MEG list closure established. The analysis is continued until an acceptable closure is reached and the total sample composition is reduced to a list of MEG compounds with their $\mu g/m^3$ effluents.

This document is based on the currently available MEG and MATE values. These are both evolving lists and, consequently, the data reduction and decision tables presented in Appendix A are not fixed.

2. LEVEL 1 SAMPLES, NATURE OF RETAINED SAMPLES AND LEVEL 1 DATA

In the phased approach to environmental assessment, Level 1 is the initial survey method by which hazardous streams are distinguished from those less hazardous or relatively innocuous. Level 1 sampling and analysis have been designed to generate semiquantitative (±3X) information about the organic and inorganic species of interest in the sampled stream so that pollutants do not go undetected. Level 1 data, therefore, consist of identified organic classes of compounds and inorganic elements. These data can then be used to prioritize the detailed and specific analysis required in Level 2.

This section discusses the types of field samples, retained prepared samples and data available from a Level 1 Environmental Assessment which can be prioritized for the MEG based Level 2 plan. Retained Level 1 samples are addressed as valid Level 2 samples. Any MEG organic categories or inorganic elements requiring a Level 2 sampling are discussed. Preliminary sampling recommendations are made in Section 4. Complete resampling, therefore, becomes unnecessary under this Level 2 approach providing the Level 1 sampling effects on quantitation are tolerable for the required environmental assessment.

2.1 LEVEL 1 SAMPLES

Level 1 samples can be separated into two distinct categories based on where they are analyzed in Level 1. These analytical categories are designated here as on-site and home-site. Logically the on-site Level 1 samples are reactive and/or volatile and are more suited for real time analyses. Retention of these materials for complex characterization would require special sampling equipment or a highly instrumented field laboratory. The home-site samples can be easily retained. Table 2-1 lists the Level 1 on-site and home-site samples and the MEG categories found in each.

The Level 1 on-site analyses, as listed in Table 2-1, lack specific tests for some MEG compounds, e.g., ozone and speciation on the C1-C7 category (Section 2.2). The Level 1 home-site samples, although they contain all MEG categories, do not contain some of the MEG compounds (Section 2.2).

Table 2-1. Level 1 Analytical Categories

Level 1 General Category	On-Site Determinations	MEG Category (Environmental Impact)
Air	• NOx	47
	• C1-C7	1, 2, 4, 5, 7, 8, 9, 10, 11, 13, 15, 24, 25, 26
	• CO_2, CO, O_2, N_2, H_2O, SO_2, H_2S	42, 47, 52, 53
	• H_2S, SO_2, COS, CH_3SH, CH_3CH_2SH, etc.	13, 53
	• Total particulate, $\mu g/m^3$ (samples retained)	(Data recorded)
Water	• PH, acidity, alkalinity, BOD, COD, dissolved oxygen, conductivity, dissolved and suspended solids, specific anions (samples retained)	(Data recorded for general water quality parameters - most analyses require 24 hour turnaround)
Solids	• Total output, kg/hour (samples retained)	(Data recorded for mass emission calculations and total particulate emissions)

Level 1 General Category	Home Site Samples	MEG Categories (Environmental Impact)
Air	• SASS components	All categories
Water	• Retained aqueous sampling, e.g., evaporation pond, cooling tower, etc.	All categories
Solids	• Retained bulk solid samples, e.g., feed materials, overflow bed materials, etc.	All categories

These home-site samples are fully characterized for inorganic elements and organic functionality. Level 2 sampling and analysis are, therefore, required for some MEG compounds (Table 2-2).

2.1.1 Level 1 Home-Site Samples

The home-site emission samples generally consist of a Level 1 catch taken with the Source Assessment Sampling System (SASS) (shown schematically in Figure 2-1) consisting of the following components:

- 1, 3, and 10 micron particulate cyclones
- Filter
- XAD-2 sorbent trap resin
- XAD-2 sorbent module condensate
- Impingers
- Probe and connecting line washings

These SASS components and the retained water and solid samples can, depending on the total quantities obtained from the Level 1 sampling effort, be retained as neat or as Level 1 prepared samples (e.g., in the case of a low particulate catch where all solids may have been prepared as organic and inorganic aliquots during Level 1 activities). Level 1 prepared samples consist of:

1. Inorganic aliquots:
    - SASS cyclones, filter particulates and XAD-2 sorbent and bulk solids dissolved in an acidic media through Parr bomb combustion over nitric acid
    - SASS impingers: H_2O_2 and APS (silver catalyzed ammonium persulfate), diluted to two liters
    - Bulk liquid samples: Cooling tower water, feed materials, etc.
2. Organic aliquots:
    - SASS cyclones, filter particulates and bulk solids extracted and concentrated in methylene chloride
    - SASS XAD-2 sorbent extracted and concentrated in methylene chloride

Table 2-2. MEG Compounds Not Contained in Level 1 Samples

Category	Compound	Rationale for Compound Loss
1. ALIPHALIC HYDROCARBONS	All < C7	On-site analysis is not compound specific. Butadienes, Pentenes, Cyclohexadiene, Acetylene, Propyne and Butyne are reactive.
2. HALOGENATED ALIPHALIC HYDROCARBON	● Methyl Iodide ● 1,1,2-Trichloroethane ● Carbon Tetrachloride ● Methyl bromide ● 1,2-Dichloroethane ● Methyl chloride ● Dichloropropane ● 1,2-Dichloro-1, 2-difluoroethane ● Dichlorodifluoromethane ● Trichlorofluoromethane ● Bromodichloromethane ● Chloroethane ● Dichloropropenes ● 1,1-Dichloroethane ● 1,2-Dichloroethane	On-site analysis is not compound specific. These are reported as C1-C7 species. Hexachlorobutadiene and Hexachlorocyclopentadiene are reactive.
4. HALOGENATED ETHERS	● Chloromethyl methyl ether ● 2-Chloroethyl methyl ether	On-site analysis is not compound specific. Chloromethyl methyl ether is reactive.

Table 2-2. MEG Compounds Not Contained In Level 1 Samples (Continued)

Category	Compound	Rationale for Compound Loss
5. ALCOHOLS	• 1-Propanol • Methanol • Ethanol • 2-Butanol • 2-Propanol • Tertiary Pentanol	On-site analysis is not compound specific.
7. ALDEHYDES, KEYTONES	• Acrolein • Formaldehyde • Propionaldehyde • Butyraldehyde • Acetaldehyde • 3-Methylbutanol • Acetone • Butanone	On-site analysis is not compound specific. Acrolein is reactive.
8. CARBOXYLIC ACIDS	• Methyl Methacrylate	This compound is reactive and may not be present in its emitted state.
9. NITRILES	• 1-Cyanoethane • Tetramethyl-succinonitrile • Butyronitrile • Benzonitrile • Acetonitrile	On-site analysis is not compound specific. 1-Cyanoethane is reactive.
10. AMINES	• Butylamines • Ethylamine • 3-Aminopropene • Ethyleneimine • Dimethylamine • Diethylamine • Ethyl methylamine	On-site analysis is not compound specific.

Table 2-2. MEG Compounds Not Contained In Level 1 Samples (Continued)

Category	Compound	Rationale for Compound Loss
11. AZO COMPOUNDS	All compounds	All azo compounds are reactive and may not be present in their emitted states.
13. MERCAPTANS, SULFIDES	• Perchloromethyl Mercaptan • Benzenthiol • 1-Anthranthiol • Methyl Disulfide	These compounds are reactive and may not be present in their emitted states.
15. BENZENE SUBSTITUTED BENZENE	• Benzene	On-site analysis is not compound specific.
17. AROMATIC NITRO COMPOUNDS	• 1-Chloro-2-Nitrobenzene • 1-Chloro-4-Nitrobenzene	These are reactive and may not be present in their emitted states.
22. FUSED NON-ALTERNATE POLYCYCLIC HYDROCARBONS	• Dicyclopentadiene	This is reactive.
24. HETEROCYCLIC NITROGEN	• Furan	On-site analysis is not compound specific.
25. HETEROCYCLIC SULFUR	• Thiophene	On-site analysis is not compound specific.
26. ORGANOMETALLICS	• Alkyl Mercury • Trimethyl Arsine	On-site analysis is not compound specific.
42. CARBON	• = CO, Carbonyl	Metal carbonyl compounds are not included in Level 1 on-site samples. They will be detected as metallic cations only.
43. SILICON	• Silane	Not included in Level 1 on-site samples.

Table 2-2. MEG Compounds Not Contained In Level 1 Samples (Continued)

Category	Compound	Rationale for Compound Loss
47. NITROGEN	• Hydrazine	Not included in Level 1 on-site samples.
48. PHOSPHORUS	• Phosphine	Not included in Level 1 on-site samples. It will be detected as total P.
49. ARSENIC	• Arsine, AsH_3	Not included in Level 1 on-site samples. It will be detected as total As.
50. ANTIMONY	• Stibine, SbH_3	Not included in Level 1 on-site samples. It will be detected as total Sb.
52. OXYGEN	• Ozone	Not included in Level 1 on-site samples.
53. SULFUR	• Sulfer Dioxide, SO_2 • Sulfer Trioxide, SO_3	SASS metallic composition catalized SO_2 indeterminately to SO_3, SO_4. (7) However, SO_2 is measured by Field Gas Chromatograph.
54. SELENIUM	• Hydrogen Selenide, H_2Se • Carbon Diselenick, CSe_2	Not included in on-site samples. These are determined as total Se.
56. FLUORINE	• HF	Not included in on-site samples. Reactive with SASS.
57. CHLORINE	• Cl_2 • HCl • $COCl_2$	Cl_2 and $COCl_2$ not included in on-site samples. HCl is reactive with SASS.
58. BROMINE	• Br_2 • HBr	Br2 and HBr are not included in on-site samples. HBr is reactive with SASS

Table 2-2. MEG Compounds Not Contained In Level 1 Samples (Continued)

Category	Compound	Rationale for Compound Loss
59. IODINE	• I_2	Not included in on-site samples.
68. CHROMIUM	• Chromium Carbonyl, $Cr(CO)_6$	Not included in on-site samples. It will be determined as total Cr.
72. IRON	• Iron Carbonyl, $Fe(CO)_5$, $Fe(CO)_9$, $Fe_3(CO)_{12}$	Not included in on-site samples. It will be determined as total Fe.
76. NICKEL	• Nickel Carbonyl, $Ni(CO)_4$	Not included in on-site samples. Decomposed above 50°C and determined as total Ni.
83. MERCURY	• HgCl	Not included in on-site samples. It will be determined as total Hg.

- Bulk liquid samples extracted and concentrated in methylene chloride
- The subsequent liquid chromatographic aliquots of these organic extracts

Level 2 environmental assessment can precede from both neat and prepared Level 1 samples. However, the obtainable Level 2 information will vary according to the sample's starting condition. Unprepared samples are preferred. (See the respective inorganic and organic Level 2 analytical schemes, Sections 5 and 6.)

2.2 NATURE OF RETAINED LEVEL 1 SAMPLES

Level 1 samples retained for any period of time can undergo species loss, chemical rearrangement, or surface/interior changes. These can result from volatilization, decomposition, free radical initiated reactions, and loss of surface coatings through sample agitation. The extent of these

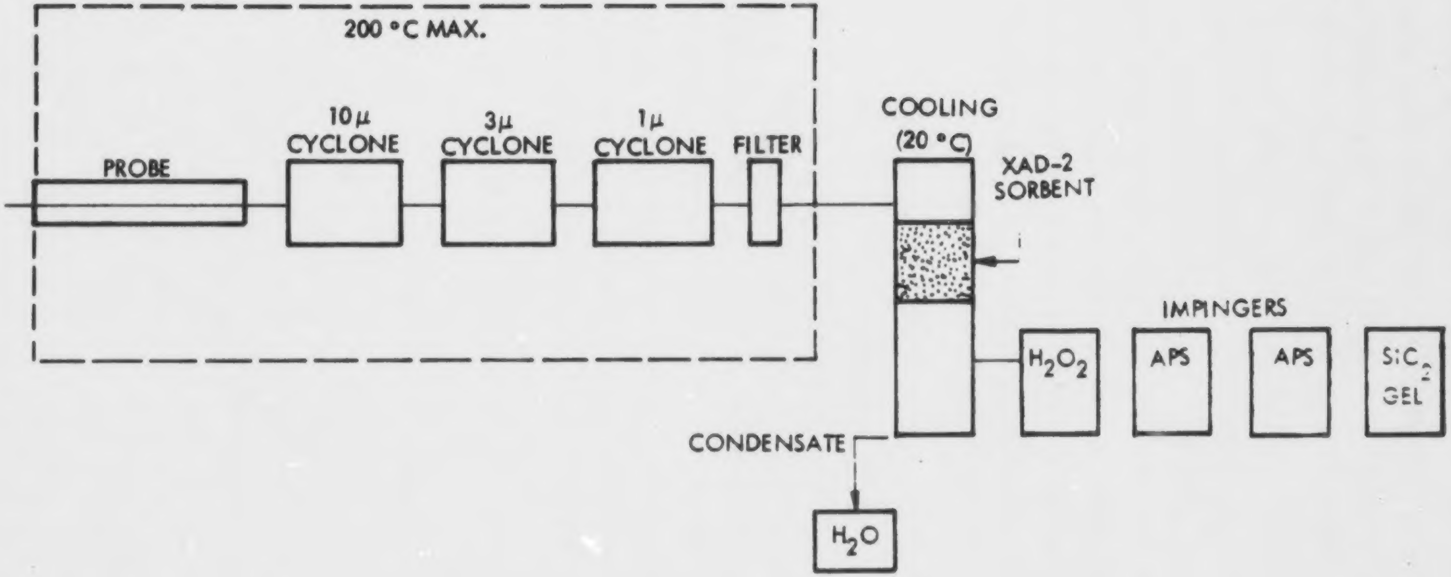

200 °C MAX.
PROBE
10μ CYCLONE
3μ CYCLONE
1μ CYCLONE
FILTER
COOLING (20 °C)
XAD-2 SORBENT
IMPINGERS
H_2O_2
APS
APS
SiO_2 GEL
CONDENSATE
H_2O

Figure 2-1. SASS Train Schematic

changes, their kinetics or their effect on the validity of generated Level 2 data are unknown. However, if the samples have been carefully stored (cool, dark, sealed, limited access storage), they should be reasonably representative of the site effluents for a period up to six months.

Retained Level 1 samples do not contain some of the MEG compounds of interest. In some cases they have not been included in the on-site sample activity; e.g., ozone; or they have reacted with the SASS contruction materials and are not sampled, e.g., HF; and in other cases, they have been sampled but altered in composition and their compound origin can no longer be distinguished, e.g., AsH_3. Table 2-2 lists the MEG categories, the compounds in each category not present or distinguishable in retained Level 1 samples, and the rationale for their absence.

Although this listing of MEG compounds not distinguishable or present in a Level 1 sampling and analysis activity is extensive, it does not impact the validity of the phased approach. The presence of most of the listed organic compounds, with the exception of 19 reactive species, is detectable in the C1-C7 as a total $\mu g/m^3$ concentration emitted for a specific boiling point range. If this total quantity exceeds the most toxic MATE value for that range, then a Level 2 sampling, followed by a compound specific analytical technique, would be initiated (Section 4). The MATE triggering values for the C1-C7 on-site samples are listed in Table 2-3.

Table 2-3. MATE Level 2 Triggering Values for C1-C6

BOILING POINT RANGE	MATE VALUE, $\mu g/M^3$	COMPOUND
C_1	3.27×10^6	Methane
C_2	5.31×10^6	Acetylene
C_3	3.3×10^2	Ethyleneimine
C_4	1.8×10^4	Ethylamine
C_5	2.5×10^2	Acrolein
C_6	3.17×10	N,N-Dimethylhydrazine

The 19 reactive organic compounds are generally dispersed throughout the MEG categories. Even though these reactive compounds will not be detected, other nonreactive compounds in these categories will trigger a Level 2 analysis because of their conservative MATE values. These 19 reactive organic compounds must have specific sampling and analysis systems designed for Level 2 on-site use. Suggested approaches for Level 2 C1-C7 and reactive organic compounds are presented in Section **4**.

Of the inorganic compounds listed, silane, ozone, hydrazine, chlorine (Cl_2), phosgene ($COCl_2$), iodine (I_2), and bromine (Br_2) are compounds not identified by Level 1 on-site sampling analyses and not collected as inorganic elements. The remaining inorganic species are detected in Level 1, but their emitted compound origin can not be retraced in home-site samples. If the Level 1 data indicate that a category, e.g., chlorine or antimony, exceeds the most conservative MATE value for any compound in that category, then Level 2 on-site activities must design protocol for these listed compounds. Suggested approaches for Level 2 on-site tests for inorganics are discussed in Section **4**.

2.3 LEVEL 1 DATA ON HOME-SITE SAMPLES

In Level 1 methodology the inorganic elements, with the exception of As, Sb, and Hg are determined by Spark Source Mass Spectroscopy (SSMS). These data are comprehensive (anion and cation information is present) and they are determined for all home-site samples. Data are reportable as $\mu g/m^3$, $\mu g/l$ and $\mu g/g$ when combined with on-site sample times, flows, and total catch data. These inorganic elemental values can easily be tabulated and compared to the MATE concentrations presented in the tables.

In the Level 1 analysis scheme, the samples are apportioned for inorganic and organic analysis. The portions for organic analysis are extracted. An aliquot is taken for the C7-C12 GC analysis, and one is taken for gravimetric and infrared (IR) analyses. If the gravimetric analysis indicates sufficient concentration of organic material, an aliquot of the extract is taken so that a minimum of 50 mg of residue results. This residue is then separated by liquid chromatography (LC). The eight LC fractions are analyzed by gas chromatography for volatiles and are evaporated for nonvolatiles, which are reportable as $\mu g/m^3$, $\mu g/l$ and $\mu g/g$ and can be compared with MATE values as tabulated in Section 3. If any fraction produces a residue that is calculated (from the sample size and volume of gas sampled) to exceed 0.5 mg/m^3, then a low resolution mass spectra (LRMS) is also obtained. For the 30 m^3 stack sample a fraction weight must exceed 15 mg. Data are then reduced to $\mu g/m^3$ for each organic SASS reporting point. The numerical criteria, which would require that a LRMS be conducted, are 0.1 mg/l for bulk water samples and 1 mg/kg for bulk solid samples.

A Level 1 IR spectrum is interpreted in terms of the presence of functional groups. That is, the presence of an absorption corresponding to a carbonyl stretch is taken as indicative of the presence of a carbonyl (categories 5, 6, 7, and 8) compound. Detection limits vary widely since the strengths of characteristic absorptions and ionization patterns vary widely in both the IR and LRMS techniques. Typically, no quantitation is performed. However, semiquantitative information is obtainable and providing that detection limits are lower than MATE requirements, MEG categories can be eliminated from consideration in the Level 2 analysis scheme.

A typical LRMS detection limit is about 1 percent of a 0.1 mg sample. Thus, about 1 μg of material should be detectable. Again providing that the detection limit in $\mu g/m^3$ for the specific MEG category on the specific instrument in use is lower than the MATE requirements, then specific categories can be eliminated from Level 2 consideration. A note can be placed on the Level 1 — Level 2 data presentation table (Appendix A) that the compound was not detectable in the Level 1 IR or LRMS.

The details of Level 1 — Level 2 decision criteria are contained in Section 3. Generally, any Level 1 reporting point (organic or inorganic) which exceeds the most conservative MATE concentration value in a given category will require Level 2 analysis on the particular Level 1 sample aliquot representative of that reporting point. Figure 2-2 depicts the Level 1 — Level 2 transition and Table 2-4 lists in general the Level 1 reporting points.

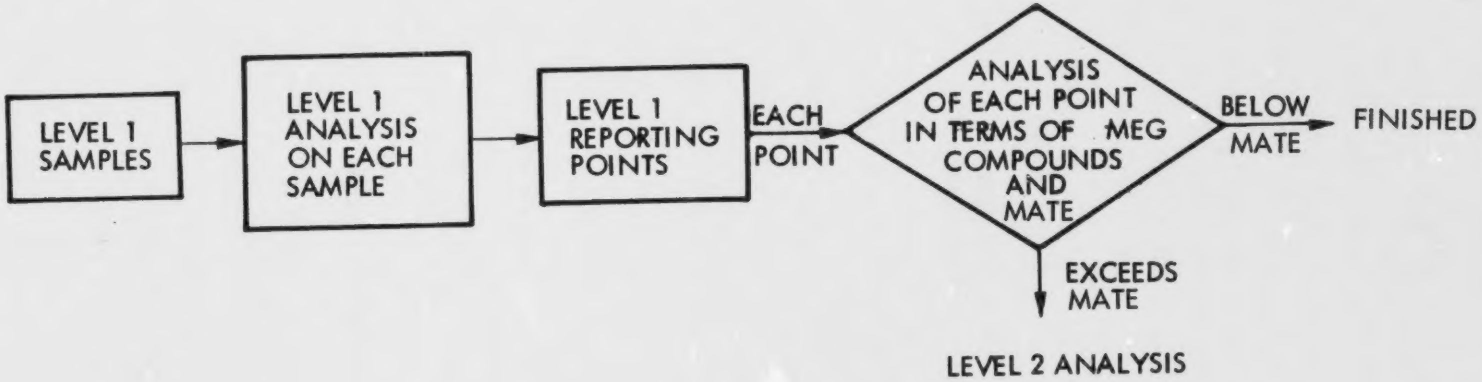

LEVEL 1
SAMPLES
LEVEL 1
ANALYSIS
ON EACH
SAMPLE
LEVEL 1
REPORTING
POINTS
EACH
POINT
ANALYSIS
OF EACH POINT
IN TERMS OF MEG
COMPOUNDS
AND
MATE
BELOW
MATE
FINISHED
EXCEEDS
MATE
LEVEL 2 ANALYSIS

Figure 2-2. Decision Logic for Phased Level 1-Level 2 Analysis

Table 2-4. General Level 1 Reporting Points

General Level 1 Sample Classes	Level 1 Reporting Point
Inorganics	● SSMS data in μg/m^3 for each element ● Hg, As, Sb data in μg/m^3
Organics	● C1-C7 on site in μg/m^3 for each boiling point range ● C7-C12 in μg/m^3 for each boiling point range ● LC1-LC8 in μg/m^3 for each MEG category
LC Fraction	MEG Category Present
1	1. Aliphatic Hydrocarbons (HCs) 2. Halogenated Aliphatic HCs
2	2. Halogenated Aliphatic HCs 15. Benzene, Substituded Benzene HCs 16. Halogenated Aromatic HCs 21. Fused Aromatic HCs 22. Fused Nonalternate Polycylic HCs
3	15. Benzene, Substituted Benzene HCs 16. Halogenated Aromatic HCs 21. Fused Aromatic HCs 22. Fused Nonalternate Polycyclic HCs 23. Heterocyclic Nitrogen Compounds

Table 2-4. General Level 1 Reporting Points (Continued)

LC Fraction	MEG Category Present
4	3. Ethers
	4. Halogenated Ethers
	9. Nitriles
	17. Aromatic Nitro Compounds
	21. Fused Aromatic HCs
	22. Fused Nonalternate Polycyclic HCs
	23. Heterocyclic Nitrogen Compounds
	25. Heterocyclic Sulfur Compounds
5	7. Aldehydes, Ketones
	9. Nitriles
	13. Mercaptans
	17. Aromatic Nitro Compounds
	18. Phenols
	24. Heterocyclic Oxygen Compounds
6	5. Alcohols
	7. Aldehydes, Ketones
	8. Carboxylic Acids, Derivatives
	9. Nitriles
	10. Amines
	18. Phenols
	19. Halophenols
7	8. Carboxylic Acids, Derivatives
	10. Amines
	11. Azo Compounds, Hydrazine Derivatives

Table 2-4. General Level 1 Reporting Points (Continued)

LC Fraction	MEG Category Present
7	18. Phenols 20. Nitrophenols
8*	8. Carboxylic Acids, Derivatives 14. Sulfuric Acid, Sulfoxides

---

*Recent studies have shown that fraction 8 does not actually contain these theoretically predicted categories. Sulfuric acid and sulfoxides may not be removed from the samples by the original extraction process.

3. MEG CATEGORIES AND COMPOUNDS AT MATE CONCENTRATION AS DECISION CRITERIA

This section presents the transitional details which start with Level 1 data and arrive at the decision to conduct Level 2 analyses. In this decision format Level 2 is only required at the specific Level 1 reporting points containing compound concentrations which exceed Minimum Acute Toxicity Effluent (MATE) guidelines. The MEG compounds, decision criteria MATE, Level 1 reporting points and generalized Level 2 tests are presented here as usable Level 1 — Level 2 data presentation tables.

3.1 MEG CATEGORIES, COMPOUNDS AND ELEMENTS

The Multimedia Environmental Goals (MEG) program concept examines the pollution potential of fossil fuel conversion processes. The results of this examination are currently presented in a listing of compounds and elements associated with coal and oil that could, based on free energies and the conversion conditions, be liberated to the environment. This is an evolving data base, changing as more biological data become available. These data include threshold limit values (TLV), median lethal dose (LD_{50}); median lethal concentrations (LC_{50}), median toxic dose (TD_{50}), and median tolerance limit (TL_m); and carcinogenic, mutagenic and teratogenic data. The biological data are then used to determine the elements and compounds targeted for control technology. The Level 1 environmental assessment methodology has been designed to identify inorganic elemental composition and organic compound categories of the sampled stream using the combination of Spark Source Mass Spectroscopy (SSMS), a sensitive (0.1 ppm) multielement analytical technique, and one element specific instrumental technique for Hg, As, and Sb. Mercury, arsenic, and antimony are volatile species with high and indeterminant limits of detection by SSMS. Organic functional groups are sought using methods such as gas chromatography, gravimetric analysis, infrared, and mass spectrometry. The inorganic or organic compounds listed in the MEG charts would not be sought by the Level 1 scheme. However, should an inorganic element or organic class exceed an EPA concentration guideline, then in the phased approach to environmental assessment, a Level 2 assessment would be required to identify and quantify the compound forms. Level 2 would be conducted to specifically seek the MEG compounds.

The MEG list provides a guide for the Level 2 analyst. The Level 2 plan developed (Sections 5 and 6) has as its basis the ability to distinguish MEG categories in the complex Level 1 samples and the overlapping Level 1 sample aliquots. The MEG compounds function as a list which the Level 2 analyst uses to produce analytical standards for verification of procedures and constantly refers to during the Level 2 analytical effort.

3.2 MATE CONCENTRATIONS

The MEG charts, as originally constructed, do not contain information on the concentration levels of interest. Concentration guidelines are necessary for the decision making process. The analyst must know the concentration at which Level 1 data trigger the Level 2 activities and the detection limits for each compound of interest in the Level 2 plan. The MATE values take into consideration a variety of factors, including the biological data listed in Section 3.1, half-lives, cumulative tendencies, and relationships between human and animal toxicity data. The MATE levels are those permissible for continuous exposure in an ambient media. Levels exceeding the MATE are, therefore, of environmental concern. MATES will be refined as more biological data and better models become available.

A decision to conduct a Level 2 analysis can now be made based on Level 1 data and MATE concentrations and their presences in a specific MEG category.

3.3 LEVEL 1 — LEVEL 2 DATA REDUCTION AND DECISION CHARTS

The data presented in Table A-1 (Appendix A) have been formulated to function for:

- Level 1 and Level 2 reporting
- Level 2 decision making based on MEG categories, compounds, and elements at MATE concentrations
- Generalizations on the applicable Level 2 techniques

The table key, which codes the analytical methodology, expectations, and cost requirements, was meant as a generalization (Sections 5 and 6 contain the specific technical analytical information). For example, GC/MS is the chosen analytical technique for all the organic categories; pretreatment, volume, GC conditions, and MS detection technique are details not meant to be part of the table key. An inorganic Level 2 Test Method example would be for soluble Fe where 1-C,A would indicate extraction (C) followed by AAS (A); another 1-C for arsenic would indicate a colorimetric determination for total As. These tables have been constructed to stand alone as decision guidelines in the Level 1 — Level 2 approach based on MEG and MATE. They list MEG compounds with their MATE in order of decreasing toxicity in each of the MEG categories, where in the Level 1 data the MEG compound of interest is found, the ratio of the specific Level 1 concentration to MATE value, if Level 2 is required, and generalizations on the applicable Level 2 methodology. A decision to conduct Level 2 tests for a specific MEG category is triggered if the Level 1 report point exceeds the most toxic MATE for that category. Level 1 bioassay results are not considered in this decision format.

4. SUGGESTED APPROACHES FOR LEVEL 2 SAMPLING AND ON-SITE ANALYSES

This section contains suggested Level 2 on-site tests for the problematic MEG compounds listed in Table 2-2. These are suggested as starting points only. Where Level 1 data have clearly indicated the requirement for Level 2 sampling and analysis then a specific sampling and analytical plan must be generated. In the on-site Level 2 plan specific hardware (e.g., all glass sampling systems, organic impinger systems, inorganic glass impingers, and particulate samplers, etc.) is to be taken back to the field along with compound specific test apparatus (e.g., nondispersive infrared analyzers, NDIR). The planning activity at that time should include specific analysis techniques and the laboratory backup, checkout and supportive data, e.g., the details of the total sample required for a successful Level 2 test.

4.1 DECISION CRITERIA FOR LEVEL 2 SAMPLING

Figure 4-1 presents a decision flow diagram to be implemented after conducting the Level 1 environmental assessment and before proceeding onto a Level 2 sampling and analysis effort. It addresses the questions the analysts must answer before establishing that the already acquired Level 1 samples are valid for Level 2. It has been designed to be a cost effective approach requiring Level 2 sampling only to acquire those data necessary for a reasonable environmental assessment. Complete resampling is not necessary.

The first criterion examines the integrity of the Level 1 tests and gathered samples. The Level 1 sampling team, home-site analytical crew, and project monitor evaluate the quality of the Level 1 samples and generated analytical data. In some cases repeat Level 1 tests would be more appropriate. Questions to be asked at this evaluation point would be:

- Was the Level 1 test statistically representative of site operating conditions?
- Has operator or instrumental error resulted in any suspect samples or data?
- Was the Level 1 representative of others conducted at similar industries and under similar conditions?

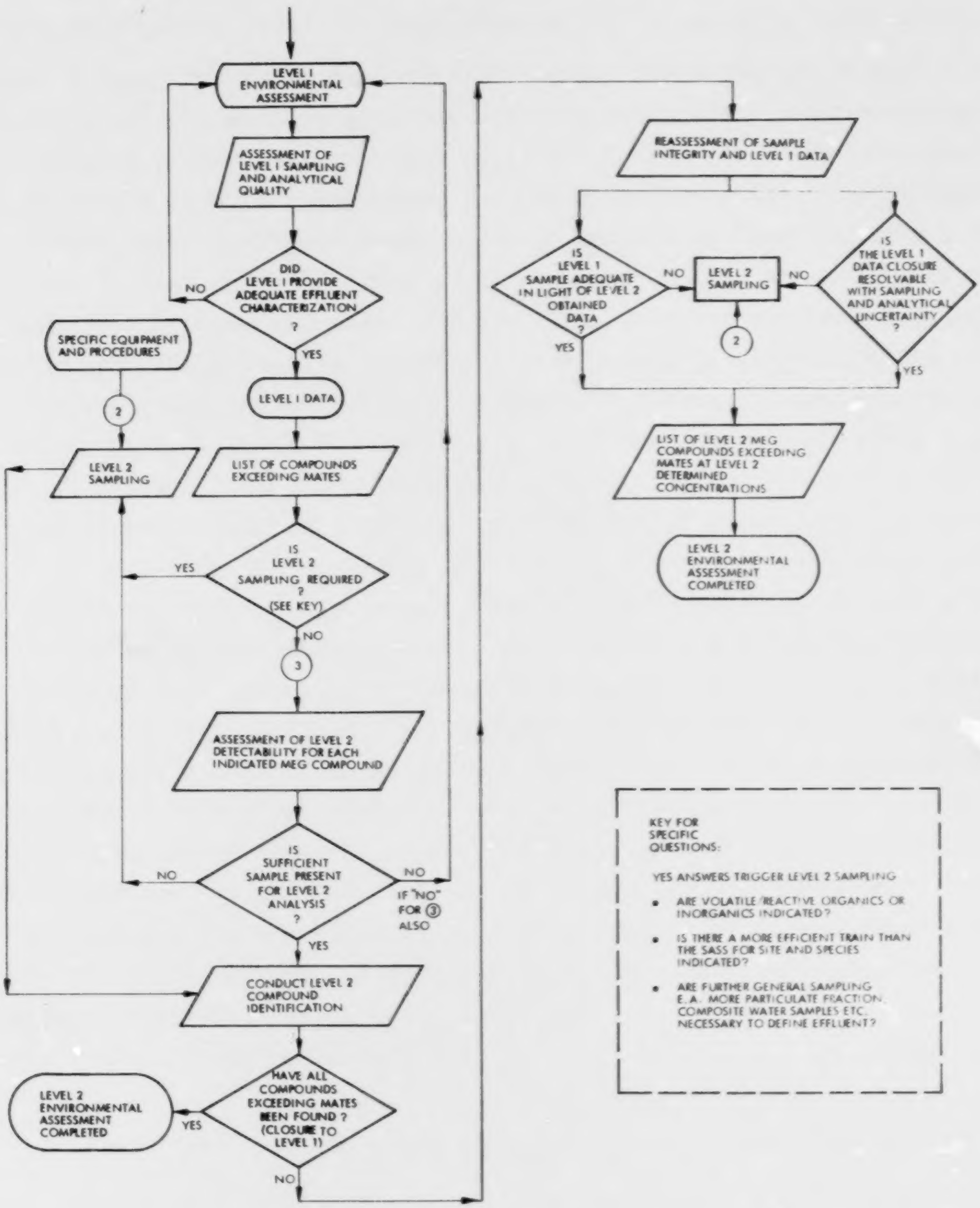

LEVEL I ENVIRONMENTAL ASSESSMENT
ASSESSMENT OF LEVEL I SAMPLING AND ANALYTICAL QUALITY
DID LEVEL I PROVIDE ADEQUATE EFFLUENT CHARACTERIZATION ?
NO
YES
SPECIFIC EQUIPMENT AND PROCEDURES
2
LEVEL I DATA
LEVEL 2 SAMPLING
LIST OF COMPOUNDS EXCEEDING MATES
IS LEVEL 2 SAMPLING REQUIRED ? (SEE KEY)
YES
NO
3
ASSESSMENT OF LEVEL 2 DETECTABILITY FOR EACH INDICATED MEG COMPOUND
IS SUFFICIENT SAMPLE PRESENT FOR LEVEL 2 ANALYSIS ?
NO
NO
IF "NO" FOR ③ ALSO
YES
CONDUCT LEVEL 2 COMPOUND IDENTIFICATION
HAVE ALL COMPOUNDS EXCEEDING MATES BEEN FOUND ? (CLOSURE TO LEVEL 1)
YES
NO
LEVEL 2 ENVIRONMENTAL ASSESSMENT COMPLETED
REASSESSMENT OF SAMPLE INTEGRITY AND LEVEL 1 DATA
IS LEVEL 1 SAMPLE ADEQUATE IN LIGHT OF LEVEL 2 OBTAINED DATA ?
NO
LEVEL 2 SAMPLING
NO
IS THE LEVEL 1 DATA CLOSURE RESOLVABLE WITH SAMPLING AND ANALYTICAL UNCERTAINTY ?
YES
2
YES
LIST OF LEVEL 2 MEG COMPOUNDS EXCEEDING MATES AT LEVEL 2 DETERMINED CONCENTRATIONS
LEVEL 2 ENVIRONMENTAL ASSESSMENT COMPLETED
KEY FOR SPECIFIC QUESTIONS:
YES ANSWERS TRIGGER LEVEL 2 SAMPLING
• ARE VOLATILE/REACTIVE ORGANICS OR INORGANICS INDICATED?
• IS THERE A MORE EFFICIENT TRAIN THAN THE SASS FOR SITE AND SPECIES INDICATED?
• ARE FURTHER GENERAL SAMPLING E.A. MORE PARTICULATE FRACTION, COMPOSITE WATER SAMPLES ETC. NECESSARY TO DEFINE EFFLUENT?

Figure 4-1. Decision Logic for Level 2 Sampling

After this data validation step, the assessment of the Level 1 chemical results can be conducted according to the decision criteria presented in Section 3 and contained on the appended Level 1 — Level 2 transition tables.

Once the probable MEG compound list has been generated, Level 2 sampling requirements are clearly discernible. For example, only volatile organic compounds (-160°C to + 100°C, the C1-C6 compounds) may require Level 2 identification and quantitation and the entire Level 2 effort would consist of a grab gas sampling followed by gas chromatography/mass spectrometry. In many cases the Level 1 samples will be suitable for the Level 2 program. Specific questions are given on Figure 4-1. When the quantity remaining may not be sufficient, then a Level 1 sampling, if less costly, can provide acceptable Level 2 samples. The analyst then follows the analytical approaches given in Sections 5 and 6. As each MEG compound triggered by Level 1 is identified and quantified, closure to the Level 1 data is checked. When successful agreement is obtained, the Level 2 assessment is completed. Some conjectural problems which the analyst could face in obtaining a reasonable closure to Level 1 involve the sample's integrity (storage changes, e.g., contamination, loses, surface oxidation) and the Level 1 uncertainty factor(quantitation factor ±3X, e.g., some inorganic elements are less reliably quantitied by the Level 1 SSMS). If the sample is contaminated to the extent that the Level 2 data obtained are scrambled, then Level 2 sampling for a higher quality sample may be more reasonable and cost effective than the identification of the interferring species. Where quantitation is problematic and multiple analyses by complimentary techniques have not resolved the closure problem, Level 2 resampling can. By employing a more efficient sampling system for the species of interest followed by the most precise analytical techniques, the analyst will resolve effluent quantitation problems. The Level 2 environmental assessment can then be reliably completed.

4.2 SUGGESTED LEVEL 2 ON-SITE TESTS FOR PROBLEMATIC MEG COMPOUNDS

The problematic MEG compounds not retained in Level 1 samples are summarized as follows:

- C1-C6 compounds, e.g., methane
- Reactive organic and inorganic compounds, e.g., acrolein and hydrogen fluoride

- Volatile inorganic compounds, e.g., phosgene
- Sampled but altered inorganic compounds, e.g., stibine

Two suggested approaches exist for C1-C6 nonreactive hydrocarbon compounds and both are complimentary:

1. Integrated Tedlar bag (glass or stainless steel sample bomb) for Level 2 resampling
    - Collect 10 liter bag sample
    - Concentrate (condense) 0.1 — 1.0 liter in laboratory
    - Conduct Gas Chromatography/Mass Spectrometry (GC/MS) using:
        a) 6 ft ss, Poropak Q
        b) 3 percent OV-101 on Chromasorb W-AW-DMCS
        c) any suitable column optimized for separation of categories 1 and 2 specifically
2. Solid absorbent method
    - Collect several 0.1 and 1.0 liter samples on a solid absorbent capable of being thermally desorbed, e.g., Tenax
    - Thermally desorb
    - Conduct GC/MS using columns suggested above

Nonreactive compounds detected in the C1-C6 range are best analyzed by direct GC/MS. Samples will need to be collected specifically for this purpose and shipment and storage should not exceed 24 hours. Two alternatives are available for these materials, and it is not possible to state a preference for one or the other at this time. (Gas sample bags and stainless steel bombs have been successfully used for direct GC/MS analysis on EPA Contract No. 68-02-2197.) A gas sample may be collected in a 10-liter bag and returned to the laboratory where 0.1 — 1.0 liter of the sample can be condensed (concentrated) in a manner similar to the Kaiser tube approach and then introduced into the GC/MS for analysis. Alternatively, several 0.1 and 1.0 liter samples can be collected on Tenax and returned to the laboratory for thermal desorption and GC/MS. This latter approach is attractive in terms of the analysis, but it is uncertain as to whether the Tenax trap is capable of retaining all of the volatile species in the C1-C6 range. Therefore, these methods are recommended as concurrent efforts.

The reactive organic compounds are best analyzed on-site as they are emitted. Some reasonable simple tests kits are available for preliminary screening during the Level 1 effort. MEG category 1 reactive compounds may also be detected in the integrated bag sample. Table 4-1 lists some reactive organic and inorganic gases which can be sampled and analyzed by use of Mine Safety Appliances (MSA) or other test kits and their range of detection. Although this is not a recommended Level 2 analysis method, it can indicate if these compounds should be considered for a more suitable Level 2 analytical method, e.g., NDIR. Judgment must be exercised with the use of these predesigned systems and interferences, reliability, and detection limits and conversion to $\mu g/m^3$ volumes made for each kit used.

Table 4-1. Reactive Organic and Inorganic Compounds Capable of Being Identified by Specific Test Kits (8)

Compound	Category	Detection Range (PPM)
Acetylene	1	3-600
Hydrazine	11 & 47	0.5-20
Mercaptans	13	0.5-100
Butanethiol	13	0.5-100
Phosphine	48	0.025-10
Arsine	49	0.025-10
Stibin	50	0.025-1.0
Ozone	52	0.05-5.0
Sulfur Dioxide	53	1-400
Hydrogen Fluoride	56	0.5-5.0
Chlorine	57	0.5-20
Hydrogen Chloride	57	2-250
Phosgene	57	0.1-10
Bromine	58	5-75
Mercury	83	0.5-2.0

NDIR (or remote FTIR which is now under development) techniques are applicable to metal carbonyls (categories 42, 68, 72, and 76), silane (category 43) and halide gases (categories 56, 57, and 58). Identification of interferences, gas conditioning requirements, and sensitivity must be assessed in the presite activities for these techniques.

Tests for the reactive organic compounds cannot be discussed in generalized terms. In the phased approach, when a category is implicated, a presite literature search, choice of analytical method and presite analytical checkout should be conducted. In these cases, as well as some inorganic areas, EPA literature and the Federal Register may contain specific test methods.

5. LEVEL 2 INORGANIC ANALYSES

Samples for Level 2 analysis may have either been retained from previous Level 1 analyses, or are new samples that have been obtained specifically in a Level 2 sampling effort. In general, the sampling systems used produce particulate matter samples from probe, cyclones, probe washings, filters, impingers, and the gas conditioner module and module condensate. Additional organic or inorganic solids, such as feed materials, both solid and liquid portions of slurries, and liquids — including leachates of selected solids, are also possible Level 2 samples.

Level 2 inorganic analysis is primarily concerned with compound identification and consequently the emphasis is placed on the analysis of solid materials. Analysis of liquid samples for inorganic material is concerned with the precise elemental composition, oxidation state of free or complexed elements, and the anion content of the sample. The following sections discuss the preparation, analysis, and correlation of data for Level 2 inorganic analysis. The approach presented here has been under development on EPA contract 68-02-2165. Preliminary details were applied and further expanded for this presentation.

5.1 INORGANIC COMPOUND IDENTIFICATION

The analysis of inorganic compounds requires the coordinated use of a variety of analytical techniques. Some techniques, such as XRD, TEM-SAED and ESCA, have the potential for direct compound identification, but only for selected compounds. The approaches described in Figures 5-1 through 5-3 are of increasing analytical complexity, designed to be cost and time effective. The identification scheme consists of:

- Initial Sample Characterization — elemental composition, sample stability, and bulk morphological structure are determined.
- Bulk Composition Characterizations — qualitative and quantitative anion, oxidation state, and X-ray diffraction information are derived.
- Individual Particle Characterization — single particle elemental composition, X-ray diffraction pattern and morphology are measured.

The degree to which each method can be applied will vary considerably with the experience, sample quantity and equipment available to the analyst. It is recommended that continued use of any one method be evaluated in light of the information derived. In general, it is far better to use a variety of instruments operated in the most efficient manner rather than pushing a single instrument or technique to the limit of its capabilities.

Figures 5-4 through 5-6 describe a logic path for identification of inorganic compounds in a solid matrix using the methods from Figure 5-1. A similar approach for liquid samples is described in Figure 5-7. In both approaches emphasis is placed on reaching an accurate closure to the MEG compounds which exceed MATE values. After a method or series of methods has been applied, a comparison is made of lists of identified to potential MEG compounds for elements which exceed their MATE values. A satisfactory analysis will depend upon a variety of factors:

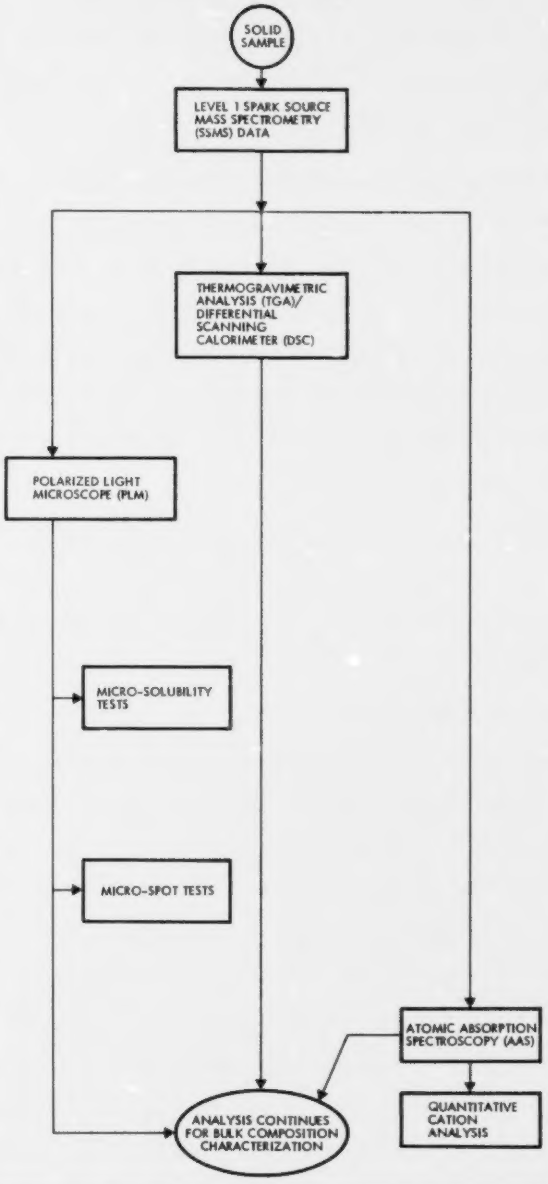
- Number of compounds identified versus MEG compounds exceeding MATE values
- Interest in identifying the remaining compounds for those elements that have exceeded MATE values
- Cost/availability of necessary equipment

The analyst must decide what method will be applied and how much more information can be obtained by each additional analysis. In many cases some methods can be bypassed because of results from previous tests, e.g., quantitative anion analysis may provide sufficient information and FTIR would only be repetitious. In other cases efforts may direct the analyst to a specific method when it would be best to analyze for a given compound. The following sections provide a discussion of the proposed methodology and information derived. By understanding the output from each technique, the analyst will be better able to select the appropriate combination of techniques to determine the compounds present in the environmental sample of interest.

5.2.1 Initial Sample Characterization

Initially, information from all sources (Level 1 field and analytical data) concerning the composition of the sample is pooled, assessed, and

ANALYSIS PROCEDURE FLOW

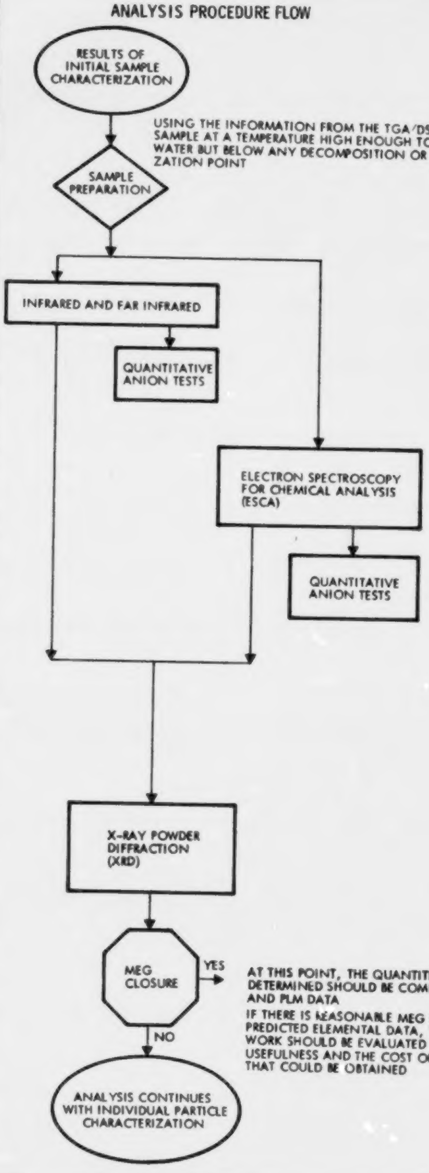

SOLID SAMPLE
LEVEL 1 SPARK SOURCE MASS SPECTROMETRY (SSMS) DATA
THERMOGRAVIMETRIC ANALYSIS (TGA)/ DIFFERENTIAL SCANNING CALORIMETER (DSC)
POLARIZED LIGHT MICROSCOPE (PLM)
MICRO-SOLUBILITY TESTS
MICRO-SPOT TESTS
ATOMIC ABSORPTION SPECTROSCOPY (AAS)
QUANTITATIVE CATION ANALYSIS
ANALYSIS CONTINUES FOR BULK COMPOSITION CHARACTERIZATION

PRINCIPLE OF OPERATION
RF POTENTIAL USED TO BREAKDOWN SAMPLE PLACED IN TWO ELECTRODES RESULTANT IONS ACCELERATED OUT OF SOURCE THROUGH ELECTROSTATIC AND ELECTROMAGNETIC ANALYZERS (SIMILAR TO ORGANIC MASS SPECTROMETRY) DETERMINE MASS DISTRIBUTION IN RESULTANT ION BEAM. USE DETECTION SYSTEM THAT PROVIDES REQUIRED SENSITIVITY AND PRECISION • PHOTOGRAPHIC PLATE SYSTEM. USED FOR TOTAL CHARACTERIZATION OF SAMPLE SINCE ENTIRE PERIODIC TABLE IS EXAMINED AND POSSIBLE INTERFERRING IONS ARE RESOLVED • ELECTRICAL DETECTION SYSTEM. GOOD FOR SINGLE ELEMENT DETERMINATION
TGA RECORDS WEIGHT LOSS OR GAIN AS MATERIAL IS HEATED DSC MEASURES HEAT EVOLVED OR ABSORBED AS SAMPLE IS HEATED
PARTICLES ARE COLLECTED FROM VARIOUS SOURCES, CRUSHED TO 0.05 MM, AND EXAMINED WITH MICROSCOPE. OF SPECIAL IMPORTANCE ARE OBSERVATIONS OF: • REFRACTIVE INDEX (RELIEF) • ISOTROPY OR ANISOTROPY • BIREFRINGENCE • PLEOCHROISM • FRACTURE • COLOR • CRYSTAL HABIT
VIEW SMALL AMOUNTS OF SAMPLE UNDER PLM WHILE ADDING COLD WATER, HOT WATER, DILUTE HCl, AND DILUTE BICARBONATE SOLUTIONS RECORD INFORMATION ON INDIVIDUAL PARTICLE SOLUBILITIES
ISOLATE SINGLE PARTICLES ON STAGE OF PLM: • MICRO-SPOT TEST FOR SPECIFIC ANIONS ($SO_4^=$, NO_2^-, NO_3^-, $CO_3^=$, Cl^-) • COMPARE TO QUANTITATIVE STANDARDS
INTRODUCE SAMPLE INTO AAS AND DECOMPOSE WITH FLAME OR HEAT OF FURNACE UNTIL GASEOUS METALLIC ATOMS ARE FORMED MAKE QUANTITATIVE DETERMINATION OF AMOUNT OF METAL IN SAMPLE BY COMPARING LEVEL OF SAMPLE ABSORBANCE AT SPECIFIC WAVELENGTHS WITH THAT OF KNOWN STANDARDS

INFORMATION DERIVED	COMPOUND IDENTIFICATION PROCEDURE	LIMITATIONS
PROVIDES ELEMENTAL CONCENTRATION DATA ON ELEMENTS CAN DETERMINE TRACE ELEMENTS IN QUANTITIES AS LOW AS 0.01 PPM ABSOLUTE SENSITIVITIES RANGE FROM 1 THROUGH 400 NG	NEED ELEMENTAL DISTRIBUTION DATA TO REDUCE COMPOUND CHOICES ELEMENTAL INFORMATION ESPECIALLY USEFUL IN INTERPRETING IR AND XRD DATA	ACCURACY OF ANALYSIS TYPICALLY 100 TO 500%. IF ONLY LEVEL 1 DATA IS USED, COULD ALLOW A YES OR NO ANSWER TO THE PRESENCE OF POSSIBLE COMPOUNDS
TGA PROVIDES SPECIFIC INFORMATION ON THERMAL STABILITY OF SAMPLE: WEIGHT LOSS CAN SOMETIMES BE CORRELATED WITH DECOMPOSITION OF SPECIFIC COMPOUNDS DSC DATA GIVES INFORMATION ON PHASE TRANSITIONS OR CHEMICAL REACTIONS IN SAMPLE	TGA/DSC NORMALLY CANNOT DETERMINE COMPOUNDS PRESENT IN COMPLEX MIXTURES WITHOUT INFORMATION ON ELEMENTAL AND ANION COMPOSITION PRIMARY USE IN THIS IDENTIFICATION SCHEME IS TO PROVIDE STABLE DRYING TEMPERATURES AND IDENTIFY ANY REACTIVE OR VOLATILE MATERIALS PRESENT	SINCE SMALL SAMPLE SIZES ARE NORMALLY USED, CHEMICAL STABILITY OF LOW CONCENTRATION MATERIALS IS NOT SEEN THE MAXIMUM TEMPERATURE OF MOST INSTRUMENTS IS 1000°C, WHICH IS BELOW DECOMPOSITION POINT OF MANY COMPOUNDS
AT LOW MAGNIFICATION, GENERAL APPEARANCE OF SAMPLE IS NOTED FOR QUALITY CONTROL OF SAMPLE HANDLING/STORAGE AT HIGHER MAGNIFICATION, CRYSTAL STRUCTURE, COLOR, REFRACTIVE INDEX ARE MEASURED FOR SINGLE PARTICLES INITIAL VIEW INDICATES MINIMUM NUMBER OF DIFFERENT PARTICLES AND THEIR POTENTIAL COMPOUNDS	ALL CRYSTALLINE COMPOUNDS HAVE SPECIFIC REFRACTIVE INDEXES WHICH CAN BE USED TO IDENTIFY THE COMPOUND AMORPHOUS MATERIALS CAN SOMETIMES BE IDENTIFIED BY COMPARISON TO KNOWN SUBSTANCES VIA PARTICLE ATLAS	LIMITED TO SINGLE PARTICLE ANALYSIS TRACE CONSTITUENTS ADSORBED ON PARTICLES OR EXTREMELY SMALL PARTICLES (0.5μ) MUST BE MEASURED WITH ANOTHER TECHNIQUE (SEM-EDX) HOMOGENEITY IMPORTANT FOR CORRECT IDENTIFICATION RESULTS DIFFICULT TO QUANTITATE
SOLUBILITY OF PARTICLES IN SPECIFIC SOLVENTS INDICATE THE CLASS OF COMPOUNDS PRESENT	USE SOLUBILITY DATA TO VERIFY LATER RESULTS USE SOLUBILITY DATA IN CONJUNCTION WITH ANION MICRO-SPOT TESTS TO REDUCE NUMBER OF POSSIBLE COMPOUND CHOICES	MICRO-TESTS ON A MICROSCOPE STAGE REQUIRE GOOD TECHNIQUE AND EXTREME CARE RESULTS REFLECT COMPOSITION OF SINGLE PARTICLES AND NOT THE BULK OF THE SAMPLE
REVEALS PRESENCE OR LACK OF SPECIFIC ANIONS	COMBINATION ON ANION AND SOLUBILITY INFORMATION LIMITS THE NUMBER OF CATIONS PRESENT IN SAMPLE AND AIDS IN SINGLE PARTICLE COMPOUND IDENTIFICATION	MICRO-TESTS ON MICROSCOPE STAGE REQUIRE GOOD TECHNIQUE AND EXTREME CARE RESULTS DIFFICULT TO QUANTITATE
PROVIDES CONCENTRATION DATA ON METALS WITH FLAMELESS TECHNIQUES, DETECTION LIMITS BETWEEN 0.001 AND 1 NG ARE POSSILBE FOR VARIOUS ELEMENTS	SPECIFIC CATIONS CAN BE IDENTIFIED AND QUANTITATED THESE METALS CAN BE CORRELATED WITH SPECIFIC PARTICLE TYPES CATION INFORMATION CAN BE COUPLED WITH SOLUBILITY AND ANION CONTENT INFORMATION TO AID IN COMPOUND IDENTIFICATION	ANALYSES OF NON-METALS AND METALLOIDS CANNOT BE PERFORMED

Figure 5-1. Initial Sample Characterization

ANALYSIS PROCEDURE FLOW


RESULTS OF INITIAL SAMPLE CHARACTERIZATION
SAMPLE PREPARATION
INFRARED AND FAR INFRARED
QUANTITATIVE ANION TESTS
ELECTRON SPECTROSCOPY FOR CHEMICAL ANALYSIS (ESCA)
QUANTITATIVE ANION TESTS
X-RAY POWDER DIFFRACTION (XRD)
MEG CLOSURE
YES
NO
ANALYSIS CONTINUES WITH INDIVIDUAL PARTICLE CHARACTERIZATION

USING THE INFORMATION FROM THE TGA/DSC, DRY THE SAMPLE AT A TEMPERATURE HIGH ENOUGH TO REMOVE WATER BUT BELOW ANY DECOMPOSITION OR VOLATILIZATION POINT

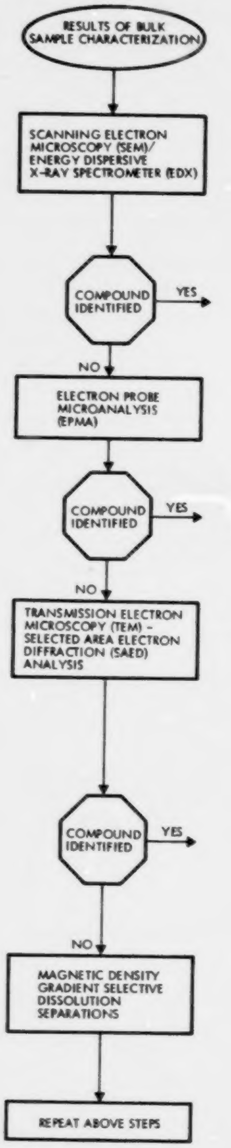
AT THIS POINT, THE QUANTITIES OF THE COMPOUNDS DETERMINED SHOULD BE COMPARED TO THE SSMS, AAS, AND PLM DATA

IF THERE IS REASONABLE MEG CLOSURE (±30%) WITH PREDICTED ELEMENTAL DATA, THE VALUE OF FURTHER WORK SHOULD BE EVALUATED IN LIGHT OF THE POTENTIAL USEFULNESS AND THE COST OF THE ADDITIONAL DATA THAT COULD BE OBTAINED

PRINCIPLE OF OPERATION	
MANY INORGANIC ANIONS HAVE SPECIFIC ABSORPTION BANDS IN INFRARED AND FAR INFRARED. THESE BANDS CAN BE USED TO IDENTIFY AND QUANTIFY THE ANIONS PRESENT EITHER WET CHEMICAL OR QUANTITATIVE IR TECHNIQUES DIRECTED TOWARD SPECIFIC ANIONS	
SAMPLE IS IRRADIATED WITH X-RAYS, CAUSING INNER SHELL ELECTRONS TO BE EJECTED: • ENERGY OF THESE EJECTED ELECTRONS IS A MEASURE OF THE BINDING ENERGY OF ELECTRONS AS MODIFIED BY THE CHEMICAL SURROUNDINGS OF THE EMITTING ATOM • ENERGY SHIFTS IN THE BINDING ENERGY OF ELECTRONS EMITTED FROM SAME ELEMENT INDICATE DIFFERENT CHEMICAL ENVIRONMENTS	
POWDER SAMPLE DIFFRACTS A PRIMARY X-RAY BEAM INTO A SERIES OF DIFFRACTION LINES CHARACTERISTIC OF A GIVEN CRYSTALLINE SUBSTANCE QUANTITATIVE COMPOUND DETERMINATIONS ARE MADE, COMMONLY USING AN INTERNAL STANDARD WITH SUBSEQUENT QUANTIFICATION BY COMPARISON TO STANDARD CURVES	

INFORMATION DERIVED	COMPOUND IDENTIFICATION PROCEDURE	LIMITATIONS
USED TO DETERMINE PRESENCE OF SPECIFIC ANIONS SUCH AS $MnO_4^{=}$, $PO_4^{\equiv}$, OR $CrO_4^{=}$ CONFIRMATION AND QUANTITATION OF SPECIFIC ANIONS	ONLY SMALL SHIFTS ARE SEEN IN THE SPECTRA WITH DIFFERENT CATIONS. ANION INFORMATION IS ESSENTIAL FOR INTERPRETING XRD DATA TO ELIMINATE POTENTIAL COMPOUNDS RATIO'S OF ANION/CATIONS USED TO PREDICT POTENTIAL COMPOUNDS	INORGANIC HALOGENS HAVE NO BANDS IN THE IR SPECTRA CAN CHANGE DEPENDING ON MOISTURE CONTENT OF SAMPLE TIME CONSUMING, SINCE DIRECTED TOWARD SPECIFIC ANION
ELEMENTAL CHARACTERIZATION DETERMINES OXIDATION STATE OF ELEMENTS PRESENT IN SAMPLE CAN DETERMINE BULK CONCENTRATIONS OF HOMOGENEOUS SAMPLES AT OR ABOVE 0.1% THOUGH ESCA IS EXTREMELY SURFACE LIMITED SINCE ELECTRONS HAVE SHALLOW (3 TO 20A$^{\circ}$) ESCAPE DEPTH, THIS MAKES THE ESCA A VERY USEFUL TOOL FOR STUDYING ABSORPTION PHENOMENA SUCH AS SO_2 ON SOOT OR FLYASH MOST COMMERCIAL INSTRUMENTS HAVE ION ($Ar+$) BEAM FOR SEQUENTIAL REMOVAL OF ATOMIC LAYERS FOR DEPTH PROFILE ANALYSIS	DIRECT COMPOUND IDENTIFICATION NOT NORMALLY POSSIBLE SINCE THERE ARE USUALLY ONLY SMALL SHIFTS IN BINDING ENERGY OF ELEMENTS IN THE SAME OXIDATION STATE ASSOCIATED WITH ANIONS OR CATIONS	INTERPRETATION AND QUANTITATION OF DATA IS DIFFICULT AND REQUIRES STANDARDS MATCHING THE MATRIX
INTERPRETATION OF DIFFRACTION PATTERN PROVIDES QUALITATIVE INFORMATION ON CRYSTALLINE MATERIALS PRESENT. DIFFRACTION LINES ARE MATCHED WITH SPECTRA OF PURE COMPOUNDS IN THE ASTM POWDER DIFFRACTION TABLES	THE DIFFRACTION LINES ARE STUDIED AND POTENTIAL COMPOUND DIFFRACTION SPECTRA ARE COMPARED TO LINES IN SAMPLE SPECTRA POTENTIAL COMPOUNDS ARE ELIMINATED OR PROPOSED BASED ON INFORMATION FROM SSMS OR AAS (ELEMENTAL DISTRIBUTION), ESCA (OXIDATION STATE), AND IR (ANIONS PRESENT)	STATE OF THE ART SENSITIVITY IS LIMITED TO $\sim$0.05% DEPENDING ON COMPOUND AND MATRIX. ROUTINE SENSITURE IS CLOSER TO 0.5% ONLY CRYSTALLINE MATERIALS CAN BE SEEN

Figure 5-2. Bulk Composition Characterization


ANALYSIS PROCEDURE FLOW
RESULTS OF BULK SAMPLE CHARACTERIZATION
SCANNING ELECTRON MICROSCOPY (SEM)/ ENERGY DISPERSIVE X-RAY SPECTROMETER (EDX)
COMPOUND IDENTIFIED
YES
NO
ELECTRON PROBE MICROANALYSIS (EPMA)
COMPOUND IDENTIFIED
YES
NO
TRANSMISSION ELECTRON MICROSCOPY (TEM) - SELECTED AREA ELECTRON DIFFRACTION (SAED) ANALYSIS
COMPOUND IDENTIFIED
YES
NO
MAGNETIC DENSITY GRADIENT SELECTIVE DISSOLUTION SEPARATIONS
REPEAT ABOVE STEPS

ANALYSIS PROCEDURE FLOW	PRINCIPLE OF OPERATION	INFORMATION DERIVED
SCANNING ELECTRON MICROSCOPY (SEM)/ ENERGY DISPERSIVE X-RAY SPECTROMETER (EDX)	IN SEM: - THE SPECIMEN IS SWEPT BY ELECTRON BEAM - SECONDARY ELECTRON EMISSION INTENSITY IS RECORDED - THE SIGNAL MODULATES BRIGHTNESS OF OSCILLOSCOPE BEAM, PRODUCING AN IMAGE - MORPHOLOGICAL CHARACTERISTICS OF THE SPECIMEN ARE DETERMINED FROM THE IMAGE USING SEM IN CONJUNCTION WITH EDX: - THE SECONDARY X-RAYS PRODUCED ARE MONITORED AND INDIVIDUAL ELEMENTS PRESENT IN THE SAMPLE ARE IDENTIFIED AND QUANTIFIED	SEM SYSTEM PROVIDES HIGH RESOLUTION MORPHOLOGICAL INFORMATION ON INDIVIDUAL PARTICLES THE EDX ATTACHMENT ALLOWS IDENTIFICATION OF INDIVIDUAL ELEMENTS IN THE PARTICLE SPECIFIC X-RAY FLUORSCENT WAVELENGTHS CAN BE MONITORED TO PRODUCE ELEMENTAL DISTRIBUTION OF THE ELEMENT (NOTE: THESE PLOTS ARE ESPECIALLY USEFUL FOR PARTICLES COMPOSED OF VARIOUS OCCLUDED MATERIALS) SEM INFORMATION IS A VALUABLE ADJUNCT TO THE PLM, ESPECIALLY FOR PARTICLES < 0.5µ (SEM MAGNIFICATIONS ARE ROUTINELY IN EXCESS OF 50,000X)
ELECTRON PROBE MICROANALYSIS (EPMA)	EPMA IS USED FOR ELEMENTS ABOVE ATOMIC NUMBER 6 SMALL ENERGETIC ELECTRON BEAM IMPINGES ON SURFACE OF SPECIMEN, CAUSING CHARACTERISTIC X-RAY EMISSIONS WHICH ARE ANALYZED BY WAVELENGTH DISPERSION TECHNIQUES FOR QUALITATIVE ANALYSIS: WAVELENGTH POSITIONS ARE USED FOR QUANTITATIVE ANALYSIS: PEAK HEIGHTS (INTENSITY RATIOS) ARE MEASURES ON BOTH THE UNKNOWN AND ON A STANDARD OF KNOWN COMPOSITION	OBTAINS SINGLE PARTICULATE ELEMENTAL COMPOSITION OF ELEMENTS FROM CARBON AND ABOVE MANY INSTRUMENTS USE WAVELENGTH DISPERSIVE X-RAY SPECTROMETERS AND CAN RESOLVE ELEMENTS S THROUGH Ni
TRANSMISSION ELECTRON MICROSCOPY (TEM) - SELECTED AREA ELECTRON DIFFRACTION (SAED) ANALYSIS	IN TEM, ELECTRON BEAM IS IMPINGED ON A THIN FILM OF SAMPLE AND THE RESULTANT TRANSMITTED ELECTRON BEAM IS OBSERVED AND RECORDED QUANTITATIVE ANALYSIS USING TEM IS SUPERIOR TO SEM BECAUSE: • SMALLER SAMPLES CAN BE OBSERVED AND IDENTIFIED • CHEMICAL SPECIES SUCH AS ASBESTOS ARE MORE RELIABLY IDENTIFIED, (TEM'S SELECTED AREA ELECTRON DIFFRACTION ANALYSIS IS MORE DEPENDABLE THAN SEM'S ELEMENTAL ANALYSIS)	PROVIDES HIGH RESOLUTION PHOTOGRAPHS PRODUCES SINGLE PARTICLE X-RAY DIFFRACTION PATTERN
COMPOUND IDENTIFIED	AT THIS POINT, IF ALL PARTICLES ARE NOT IDENTIFIED, THE ANALYST MUST DECIDE WHETHER FRACTIONATION OF THE SAMPLE WOULD IMPROVE THE IDENTIFICATION OF MATERIALS PRESENT IF SEPARATION STEP IS EMPLOYED, EXTREME CARE MUST BE TAKEN TO PREVENT SAMPLE ALTERATION OR COMPOUND SCRAMBLING	
MAGNETIC DENSITY GRADIENT SELECTIVE DISSOLUTION SEPARATIONS	MAGNETIC SEPARATION IS USED TO REMOVE ANY MAGNETIC MATERIAL FROM REST OF SAMPLE IN DENSITY GRADIENT SEPARATIONS, PARTICLES ARE FLOATED IN SOLVENTS OF KNOWN DENSITY. PARTICLES ARE SEPARATE BY DIFFERENCES IN THEIR DENSITY SAMPLE IS EXTRACTED USING SELECTIVE DISSOLUTION (WITH DIFFERENT SOLVENTS)	DETERMINES PARTICLES SPECIFIC DENSITY, MAGNETIC CHARACTERISTICS, AND SOLUBILITY IN SOLVENTS MAIN USE IS TO REDUCE COMPLEX SYSTEMS

	COMPOUND IDENTIFICATION PROCEDURE	LIMITATIONS
	SEM'S HIGH RESOLUTION IMAGES OFTEN ALLOW PARTICLE IDENTIFICATION THE EDX INFORMATION CAN BE USED TO DETERMINE ELEMENTAL RATIOS AND THE EXACT COMPOSITION OF THE PARTICLE	RELATIVELY LONG COUNTING TIMES ARE REQUIRED FOR TRACE ELEMENTS, BUT THE EDX INSTRUMENT STABILITY LIMITS COUNTING TIME TO 10 OR 15 MINUTES AT HIGH COUNT RATES, PEAKS MAY BROADEN PARTICLES IN CLOSE PROXIMITY MAY INTERFERE AND PRECLUDE UNAMBIGUOUS ANALYSIS EDX DOES NOT RESOLVE ELEMENTS FROM S TO Ni VERY WELL QUANTITATIVE WORK DEPENDS ON HAVING SUITABLE STANDARDS
	COMPOUND IDENTIFIED BY ELEMENTAL RATIOS EPMA ESSENTIAL WHEN ELEMENTS C THROUGH Na ARE PRESENT SINCE SEM-EDX DOES NOT SEE THOSE ELEMENTS	IDENTIFICATION POSSIBLE ONLY FOR PARTICLES CONTAINING DISCRETE COMPOUNDS RATHER THAN A HOMOGENEOUS MIXTURE BETTER QUANTITATIVE RESULTS WHEN STANDARDS ARE USED WHOSE COMPOSITION CLOSELY MATCHES THE SPECIMEN
	IDENTIFIES CRYSTALLINE COMPOUNDS BY THEIR CHARACTERISTIC DIFFRACTION PATTERNS	ONLY CRYSTALLINE MATERIAL CAN BE IDENTIFIED
	SEPARATING COMPLEX MIXTURE INTO SIMPLER FRACTIONS AIDS COMPOUND IDENTIFICATION CAN USE INFORMATION ON PARTICLE DENSITY, SOLUBILITY, AND MAGNETIC PROPERTIES TO IDENTIFY COMPOUNDS	DENSITY GRADIENT WILL ONLY SEPARATE DISCRETE PARTICLES; OCCLUDED MATERIAL WILL HAVE AN AVERAGE DENSITY MANY COMPOUNDS HAVE SOLUBILITIES IN ORGANIC SOLVENTS USED IN DENSITY COLUMN SELECTIVE DISSOLUTION SCRAMBLES THE COMPOUNDS UNLESS SPECIFIC COMPOUND SOLVENT SYSTEMS CAN BE FOUND

Figure 5-3. Individual Particle Characterization

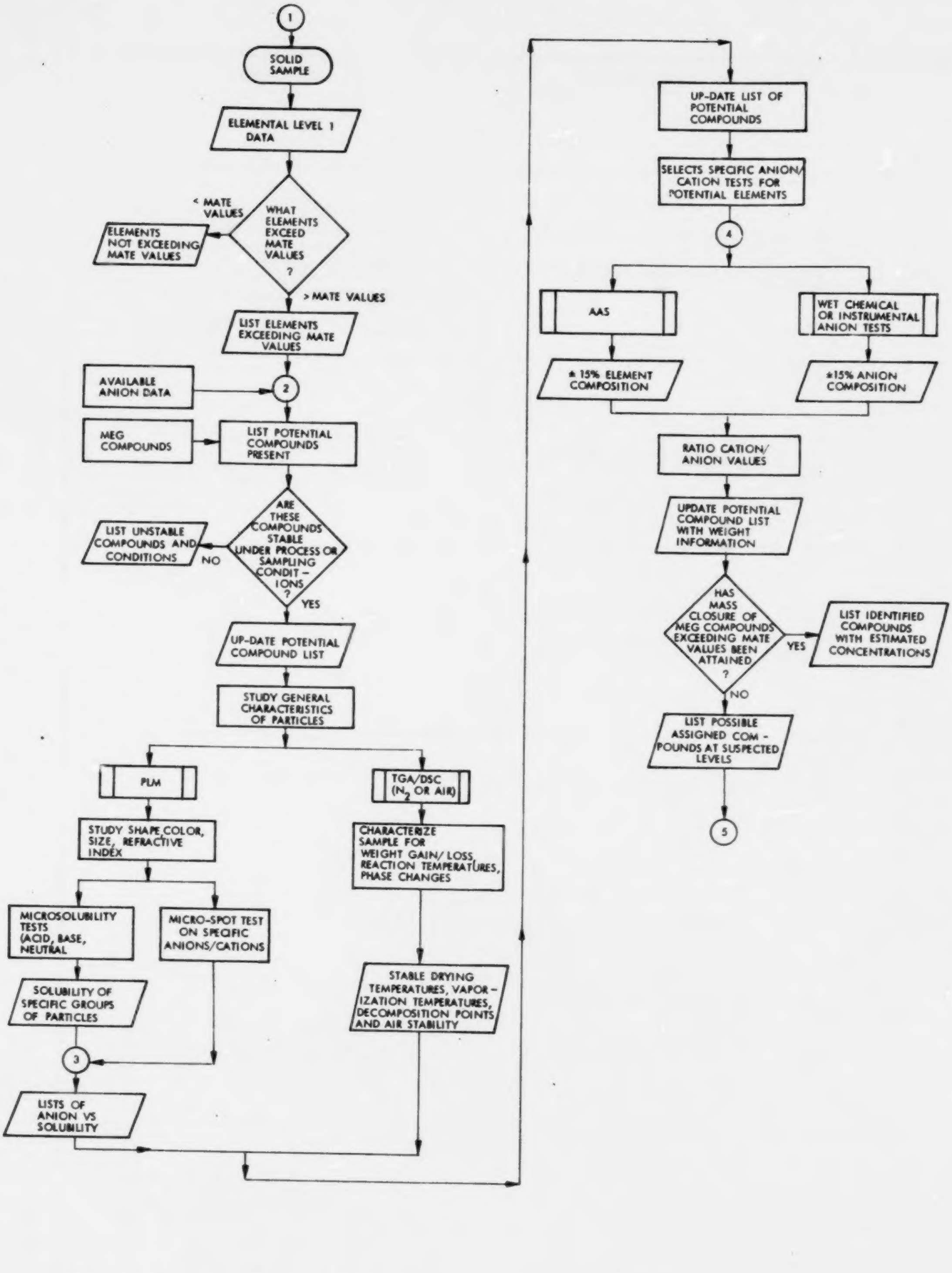

1
SOLID SAMPLE
ELEMENTAL LEVEL 1 DATA
< MATE VALUES
WHAT ELEMENTS EXCEED MATE VALUES ?
ELEMENTS NOT EXCEEDING MATE VALUES
> MATE VALUES
LIST ELEMENTS EXCEEDING MATE VALUES
AVAILABLE ANION DATA
2
MEG COMPOUNDS
LIST POTENTIAL COMPOUNDS PRESENT
ARE THESE COMPOUNDS STABLE UNDER PROCESS OR SAMPLING CONDIT-IONS ?
LIST UNSTABLE COMPOUNDS AND CONDITIONS
NO
YES
UP-DATE POTENTIAL COMPOUND LIST
STUDY GENERAL CHARACTERISTICS OF PARTICLES
PLM
TGA/DSC (N_2 OR AIR)
STUDY SHAPE,COLOR, SIZE, REFRACTIVE INDEX
CHARACTERIZE SAMPLE FOR WEIGHT GAIN/LOSS, REACTION TEMPERATURES, PHASE CHANGES
MICROSOLUBILITY TESTS (ACID, BASE, NEUTRAL
MICRO-SPOT TEST ON SPECIFIC ANIONS/CATIONS
SOLUBILITY OF SPECIFIC GROUPS OF PARTICLES
STABLE DRYING TEMPERATURES, VAPOR-IZATION TEMPERATURES, DECOMPOSITION POINTS AND AIR STABILITY
3
LISTS OF ANION VS SOLUBILITY
UP-DATE LIST OF POTENTIAL COMPOUNDS
SELECTS SPECIFIC ANION/CATION TESTS FOR POTENTIAL ELEMENTS
4
AAS
WET CHEMICAL OR INSTRUMENTAL ANION TESTS
± 15% ELEMENT COMPOSITION
±15% ANION COMPOSITION
RATIO CATION/ANION VALUES
UPDATE POTENTIAL COMPOUND LIST WITH WEIGHT INFORMATION
HAS MASS CLOSURE OF MEG COMPOUNDS EXCEEDING MATE VALUES BEEN ATTAINED ?
YES
LIST IDENTIFIED COMPOUNDS WITH ESTIMATED CONCENTRATIONS
NO
LIST POSSIBLE ASSIGNED COM-POUNDS AT SUSPECTED LEVELS
5

Figure 5-4. Logic Flow Chart for Initial Sample Characterization

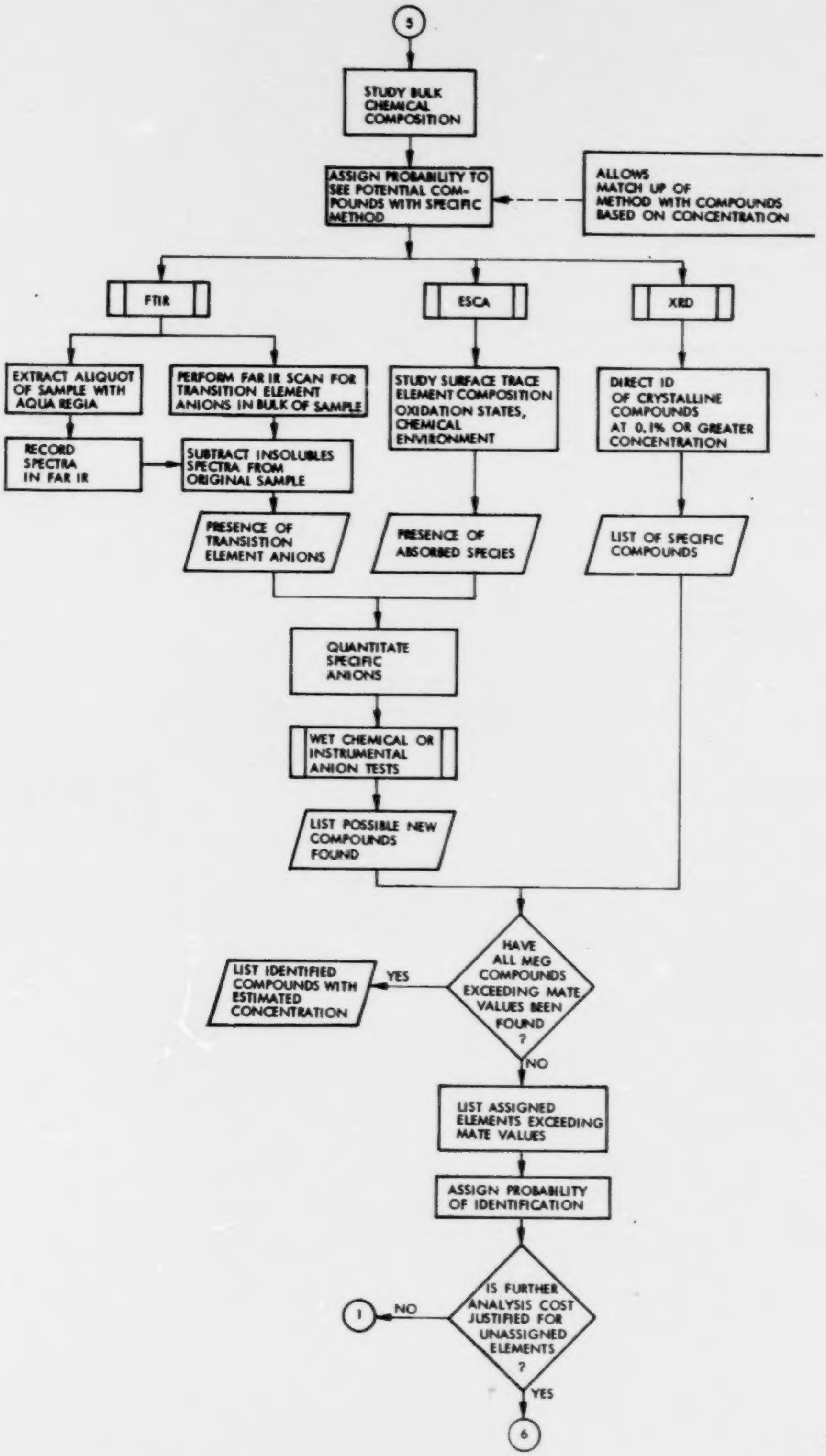

5
STUDY BULK CHEMICAL COMPOSITION
ASSIGN PROBABILITY TO SEE POTENTIAL COMPOUNDS WITH SPECIFIC METHOD
ALLOWS MATCH UP OF METHOD WITH COMPOUNDS BASED ON CONCENTRATION
FTIR
ESCA
XRD
EXTRACT ALIQUOT OF SAMPLE WITH AQUA REGIA
PERFORM FAR IR SCAN FOR TRANSITION ELEMENT ANIONS IN BULK OF SAMPLE
STUDY SURFACE TRACE ELEMENT COMPOSITION OXIDATION STATES, CHEMICAL ENVIRONMENT
DIRECT ID OF CRYSTALLINE COMPOUNDS AT 0.1% OR GREATER CONCENTRATION
RECORD SPECTRA IN FAR IR
SUBTRACT INSOLUBLES SPECTRA FROM ORIGINAL SAMPLE
PRESENCE OF TRANSISTION ELEMENT ANIONS
PRESENCE OF ABSORBED SPECIES
LIST OF SPECIFIC COMPOUNDS
QUANTITATE SPECIFIC ANIONS
WET CHEMICAL OR INSTRUMENTAL ANION TESTS
LIST POSSIBLE NEW COMPOUNDS FOUND
HAVE ALL MEG COMPOUNDS EXCEEDING MATE VALUES BEEN FOUND ?
YES
LIST IDENTIFIED COMPOUNDS WITH ESTIMATED CONCENTRATION
NO
LIST ASSIGNED ELEMENTS EXCEEDING MATE VALUES
ASSIGN PROBABILITY OF IDENTIFICATION
IS FURTHER ANALYSIS COST JUSTIFIED FOR UNASSIGNED ELEMENTS ?
NO
1
YES
6

Figure 5-5. Logic Flow for Bulk Composition Characterization

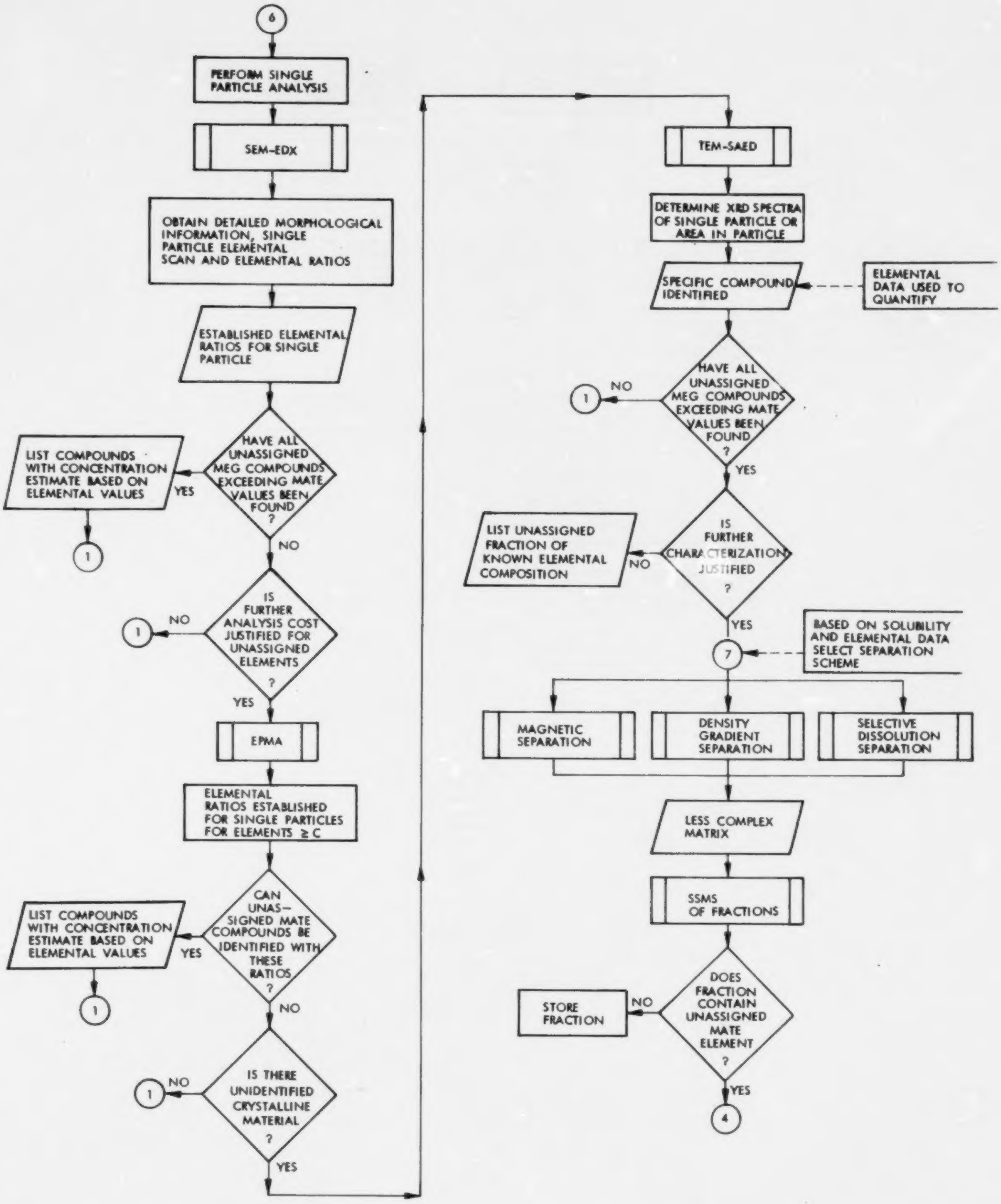

6
PERFORM SINGLE PARTICLE ANALYSIS
SEM-EDX
OBTAIN DETAILED MORPHOLOGICAL INFORMATION, SINGLE PARTICLE ELEMENTAL SCAN AND ELEMENTAL RATIOS
ESTABLISHED ELEMENTAL RATIOS FOR SINGLE PARTICLE
HAVE ALL UNASSIGNED MEG COMPOUNDS EXCEEDING MATE VALUES BEEN FOUND ?
YES
LIST COMPOUNDS WITH CONCENTRATION ESTIMATE BASED ON ELEMENTAL VALUES
1
NO
IS FURTHER ANALYSIS COST JUSTIFIED FOR UNASSIGNED ELEMENTS ?
NO
1
YES
EPMA
ELEMENTAL RATIOS ESTABLISHED FOR SINGLE PARTICLES FOR ELEMENTS ≥ C
CAN UNAS-SIGNED MATE COMPOUNDS BE IDENTIFIED WITH THESE RATIOS ?
YES
LIST COMPOUNDS WITH CONCENTRATION ESTIMATE BASED ON ELEMENTAL VALUES
1
NO
IS THERE UNIDENTIFIED CRYSTALLINE MATERIAL ?
NO
1
YES
TEM-SAED
DETERMINE XRD SPECTRA OF SINGLE PARTICLE OR AREA IN PARTICLE
SPECIFIC COMPOUND IDENTIFIED
ELEMENTAL DATA USED TO QUANTIFY
HAVE ALL UNASSIGNED MEG COMPOUNDS EXCEEDING MATE VALUES BEEN FOUND ?
NO
1
YES
IS FURTHER CHARACTERIZATION JUSTIFIED ?
NO
LIST UNASSIGNED FRACTION OF KNOWN ELEMENTAL COMPOSITION
YES
7
BASED ON SOLUBILITY AND ELEMENTAL DATA SELECT SEPARATION SCHEME
MAGNETIC SEPARATION
DENSITY GRADIENT SEPARATION
SELECTIVE DISSOLUTION SEPARATION
LESS COMPLEX MATRIX
SSMS OF FRACTIONS
DOES FRACTION CONTAIN UNASSIGNED MATE ELEMENT ?
NO
STORE FRACTION
YES
4

Figure 5-6. Logic Flow for Individual Particle Characterization

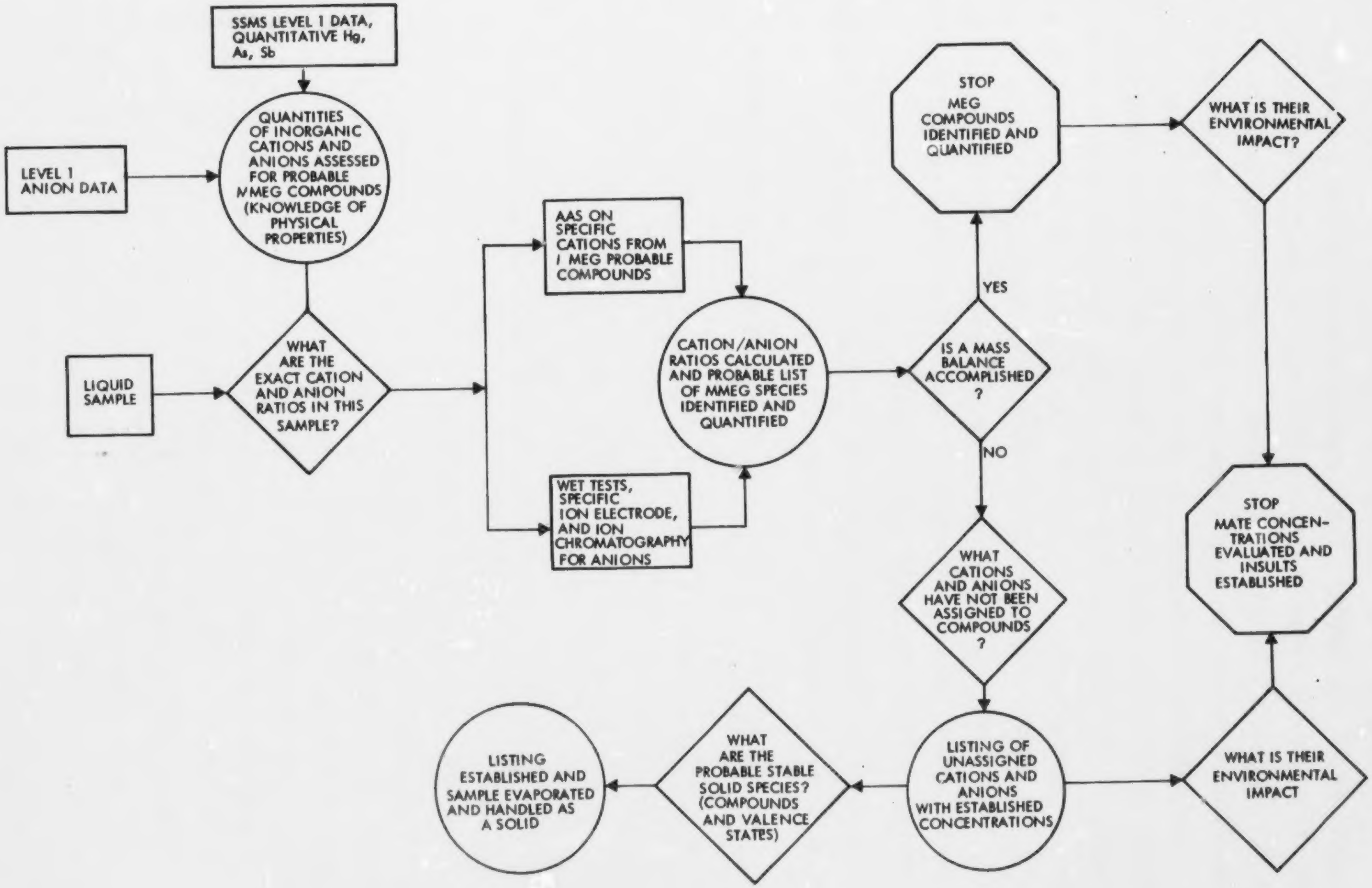

LEVEL 1 ANION DATA
SSMS LEVEL 1 DATA, QUANTITATIVE Hg, As, Sb
QUANTITIES OF INORGANIC CATIONS AND ANIONS ASSESSED FOR PROBABLE MMEG COMPOUNDS (KNOWLEDGE OF PHYSICAL PROPERTIES)
LIQUID SAMPLE
WHAT ARE THE EXACT CATION AND ANION RATIOS IN THIS SAMPLE?
AAS ON SPECIFIC CATIONS FROM / MEG PROBABLE COMPOUNDS
WET TESTS, SPECIFIC ION ELECTRODE, AND ION CHROMATOGRAPHY FOR ANIONS
CATION/ANION RATIOS CALCULATED AND PROBABLE LIST OF MMEG SPECIES IDENTIFIED AND QUANTIFIED
IS A MASS BALANCE ACCOMPLISHED ?
YES
NO
STOP MEG COMPOUNDS IDENTIFIED AND QUANTIFIED
WHAT IS THEIR ENVIRONMENTAL IMPACT?
WHAT CATIONS AND ANIONS HAVE NOT BEEN ASSIGNED TO COMPOUNDS ?
LISTING OF UNASSIGNED CATIONS AND ANIONS WITH ESTABLISHED CONCENTRATIONS
WHAT ARE THE PROBABLE STABLE SOLID SPECIES? (COMPOUNDS AND VALENCE STATES)
LISTING ESTABLISHED AND SAMPLE EVAPORATED AND HANDLED AS A SOLID
WHAT IS THEIR ENVIRONMENTAL IMPACT
STOP MATE CONCENTRATIONS EVALUATED AND INSULTS ESTABLISHED

Figure 5-7. Level 2 Liquid Sample Compound Analysis Scheme

used for reference. This information provides the first insight into the composition of the sample. The elemental concentrations are compared to MATE values to determine which compounds must be sought (Section 3). Once the MEG compounds exceeding MATE values are identified, then the elemental composition data will be used by the analyst to determine whether or not specific compounds are present, as well as indicating whether a given method has sufficient sensitivity to detect the potential compound. For example, if the SSMS data show that a given element is present at levels above 0.5 percent, then bulk analysis by XRD might be successful in determining the compound form of this element. Elemental information is especially important to XRD because diffraction patterns of environmental samples are complex and information reducing the potential possibilities is necessary for a cost effective analysis.

Besides the SSMS data, there are several other sources of information that should be assembled. This information includes:

- Source of the sample: process type, e.g., oxidizing or reducing conditions.
- Composition of feed source: input raw materials, e.g., coal, limestone.
- Previous history of sample: age, storage conditions, collection method.
- Results of previous analyses at this source: elemental and compound information.

Concurrent with assembling the Level 1 data, preliminary characterization of the sample should be initiated. As soon as the sample is in the laboratory, it should be viewed under a polarized light microscope (PLM) and a photomicrograph taken in color to serve as quality control. If any changes in the general appearance of the sample occur during the duration of the analytical activities, these should be noted. The PLM can also provide a measure of the complexity of the sample simply by noting the number of different types of particles.

Polarized light microscopy is the first direct compound analysis method. Particles can be identified by the determination of such properties as the refractive index, isotropy or anisotropy, birefringence, pheochroism, fracture, color, and crystal habit. Microspot tests for common anions and tests of the solubility of the particles in water, acid, and base can be performed directly on the sample as it is being examined under the microscope. These microtests will alert the analyst to perform quantitative analyses for the anions detected and they will also provide information about the potential success of full scale dissolutions and separation.

At this stage in the analysis, quantitative analysis of anions identified in the microspot tests will most probably be performed using classical wet test methods, e.g., titrimetric, colorimetric, or specific ion electrode tests. A summary of the Level 1 and 2 analytical methods used for the more common anions is presented in Table 5-1. It is meant to serve as a starting point for the analyst, as other methods may be substituted for those suggested. Level 2 anion methods can be chosen by the analyst from Standard Methods (Water and Wastewater), ASTM, or other EPA procedures.

Also, during this initial sample characterization, the analyst may choose to supplement the SSMS semiquantitative cation data by analyzing fractions of the samples using such quantitative techniques as atomic absorption spectrometry (either flame or flameless), Induction Coupled Plasma Optical Emission Spectroscopy (ICPOES), Proton Induced X-Ray Emission (PIXE), or X-ray fluorometry.

In conjunction with the PLM work a TGA/DSC scan of the sample should be made. This test is used primarily to determine 1) the stability of the sample, and 2) an appropriate temperature at which to dry samples to be used in later tests. In a few cases it is possible to determine the compounds present by the weight loss at specific temperatures. Elemental information from SSMS and anion information from PLM (and later IR) can be combined to give a list of potential compounds that exhibit decomposition points at the weight loss points in the TGA or the exotherms and endotherms of the DSC.

At the end of the initial sample characterization, information will have been obtained in the following areas:

1. General appearance of a sample
2. Number of different particles present
3. Index of refraction and crystal structure

Table 5-1. Summary of Recommended Procedures for Anion Analysis

ANALYSIS AREA	LEVEL 1				LEVEL 2				REMARKS (LEVELS 1 AND 2)
	RECOMMENDED ANALYTICAL METHOD	SELECTION RATIONALE	SPECIAL SAMPLING AND ANALYTICAL REQUIREMENTS	REFERENCES	RECOMMENDED ANALYTICAL METHOD	SELECTION RATIONALE	SPECIAL SAMPLING AND ANALYTICAL REQUIREMENTS	ASTM	
Ammonia	Colorimetric	Rapid and simple method.	As specified by reagent test kit		Colorimetric (0.05-1 ppm) Titrimetric 1.0-25 ppm)	Method provides an accurate technique for analysis of ammonia in water. EPA method. Standard Method 132.	Sample is buffered at pH 9.5 and distilled into a solution of boric acid. The ammonia in the distillate can be determined colorimetrically by nesslerization (HgI_2, KI, NaOH) or titrimetrically with H_2SO_4 using a mixed indicator (methyl red/ methylene blue).	D1426	Volatile organic alkaline compounds may cause an off color in the nesslerization procedure.
Arsenate/ Arsenite	SSMS/AAS	Elemental analysis provides upper concentration limit for anions. SSMS has multi-component capability.	Aqueous samples: slurry with graphite and briquette. Solid samples: see element analysis. Level 2 technique is applied to most Level 1 samples.		Spectrometric	Method provides an accurate, fairly rapid technique for measuring arsenic. ASTM method.	25 ml of sample is acidified with HCl, mixed with KI and $SnCl_2$. 3 g of zinc are added and the arsine generated is bubbled through a silver diethyldithiocarbamate-pyridine solution. Absorbance is measured at 540 nm within 30 minutes.	D2972	Measurement of arsenic by AAS is also an acceptable technique and may be subject to fewer interferences.
Bromide	SSMS	As for arsenate	As for arsenate		Titrimetric	Method provides an accurate, fairly rapid technique for measuring bromide. ASTM method.	Sufficient NaCl is added to 100 ml of the sample to produce a 3g chloride content. KClO is added to oxidize bromide to bromine (excess is destroyed with $NaCHO_2$). KI is added and liberated I_2 is titrated with 0.01N $Na_2S_2O_3$.	D1246	This method measures bromide and iodide: thus, this method is to be used in conjunction with iodide determination. Fe^{+2}, Mn^{+2} interfere, but may be removed by treatment with CaO.
Carbonate (Bicarbonate)	Titrimetric	Method provides a rapid, and simple analysis technique.	As specified by reagent test kit.		Titrimetric	Method provides an accurate technique for measuring total carbonate present. ASTM method.	Apparatus is described in reference. CO_2 is liberated by acidifying and heating the sample in a closed system. CO_2 is absorbed in a barium hydroxide solution. Excess barium hydroxide is titrated with 0.04 N HCl.	D513	Sulfides, (H_2S) interfered but are removed by scrubbing with iodine solution; other interferences are removed by scrubbing with chromic acid. From pH measurement H_2CO_3, HCO_3^-, CO_3^{-2} concentrations may be estimated.

Table 5-1. Summary of Recommended Procedures for Anion Analysis (Continued)

ANALYSIS AREA	LEVEL 1				LEVEL 2				REMARKS (LEVELS 1 AND 2)
	RECOMMENDED ANALYTICAL METHOD	SELECTION RATIONALE	SPECIAL SAMPLING AND ANALYTICAL REQUIREMENTS	REFERENCES	RECOMMENDED ANALYTICAL METHOD	SELECTION RATIONALE	SPECIAL SAMPLING AND ANALYTICAL REQUIREMENTS	ASTM	
Chloride	SSMS	As for arsenate.	As for arsenate.		Titrimetric	Method provides an accurate technique for measuring chloride content of industrial wastewater. ASTM method.	50 ml sample is titrated with 0.025 N silver nitrate to a potassium chromate endpoint. Sulfites are oxidized to sulfates by H_2O_2 addition.	D512	Phosphates (>250 ppm) interfere. Iodine and bromide may also interfere with visual endpoint; potentiometric titration may solve this problem.
Cyanide	Colorimetric	Method provides a rapid, simple analysis technique.	As specified by reagent test kit.		Titrimetric/ Spectrometric	Method provides an accurate technique for measuring cyanide. ASTM method.	500 ml of sample is refluxed under acidic conditions with $CuCl_2$. HCN liberated is absorbed in NaOH. Titration: titration with $AgNO_3$ to rhodamine endpoint. Spectrometric: neutralize absorption solution with acetic acid to pH 6.5 - 8.0 0.2 ml of chloramine T solution is added. Absorbance measured at 620 nm after 20 minutes.	D2036	Titration method applies when cyanide concentration >1 ppm; spectrometric method for <1 ppm.
Fluoride	SSMS	As for arsenate.	As for arsenate.		Specific Ion Electrode (SIE)	Method provides an accurate, rapid, simple technique for analysis of fluoride.	pH is adjusted to 5.2 - 5.5 with 0.5 N H_2SO_4. CO_2 is removed by heating on a hot water bath. Buffer is added (pH 6.3, 1M sodium citrate - citric acid - 0.2 M KNO_3) and fluoride is measured by known addition method.	SIE 9	Selective ion electrode (SIE) is more accurate and simpler than distillation - spectrometric method (SPADNS).
Iodide	SSMS	As for arsenate.	As for arsenate.		Spectrometric	Method provides an accurate technique for analysis of iodide. ASTM method.	Iodide is determined by oxidation to iodate with saturated bromine water in acid solution. Excess bromine is destroyed by addition of sodium formate. Sample is titrated with 0.01 N sodium thiosulfate solution.	D1246	Effects of Fe^{+3}, Mn^{+2}, and organic matter are removed by treatment with CaO. This method is used in conjunction with bromide determination.

Table 5-1. Summary of Recommended Procedures for Anion Analysis (Continued)

ANALYSIS AREA	LEVEL 1				LEVEL 2				REMARKS (LEVELS 1 AND 2)
	RECOMMENDED ANALYTICAL METHOD	SELECTION RATIONALE	SPECIAL SAMPLING AND ANALYTICAL REQUIREMENTS	REFERENCES	RECOMMENDED ANALYTICAL METHOD	SELECTION RATIONALE	SPECIAL SAMPLING AND ANALYTICAL REQUIREMENTS	ASTM	
Nitrate	Colorimetric	Method provides a rapid and simple analysis technique.	As specified by reagent test kit.		Spectrometric	Method provides an accurate technique for analysis of nitrate. ASTM method.	5 ml of sample is mixed with brucine-sulfanilic acid solution then mixed with 10 ml of 15.6 M H_2SO_4. Color is developed for 10 ±1 minute in a dark area; absorbance is measured at 410 nm.	D992	Color does not follow Beer-Lambert relation; however, plotting absorbance vs. concentration yields a smooth curve. Turbid or colored samples interfere, but may be removed by filtration and treatment with Al_2O_3 and activated carbon.
Nitrite	Colorimetric	Method provides a rapid, simple analysis technique.	As specified by reagent test kit.		Spectrometric	Method provides an accurate technique for analysis of nitrite. ASTM method.	pH is adjusted to 7 with CH_3COOH. If sample has appreciable color, filter with $Al(OH)_3$ gel. EDTA is added to complex cations. 2 ml of sulfanilic acid solution and 2 ml of α naphthylamine hydrochloride are added to the sample, solution is buffered at pH 2.0 - 2.5 with $Na_2C_2H_3O_2$ solution, allowed to stand 30 minutes, and absorbance measured at 515 nm.	D1254	Mercury (II) causes high results while copper (II) catalyzes the decomposition of the diazonium salt and thus leads to low results. Certain bacteria utilize nitrites in their metabolism. Storage at low temperature minimizes this effect.
Ortho-Phosphate	Colorimetric	Method provides a rapid, simple analysis technique.	As specified by reagent test kit.		Spectrometric	Method provides an accurate technique for analysis of phosphate.	If pH >7, sample is neutralized with H_2SO_4. Molybdate reagent and stannous chloride reagent are added. Absorption is measured at 690 nm between 10-12 minutes after reagent addition.	D515	Color intensity is time and temperature dependent. Solution may be extracted with benzene-isobutanol solvent to remove interferences and increase sensitivity.

Table 5-1. Summary of Recommended Procedures for Anion Analysis (Continued)

ANALYSIS AREA	LEVEL 1				LEVEL 2				REMARKS (LEVELS 1 AND 2)
	RECOMMENDED ANALYTICAL METHOD	SELECTION RATIONALE	SPECIAL SAMPLING AND ANALYTICAL REQUIREMENTS	REFER-ENCES	RECOMMENDED ANALYTICAL METHOD	SELECTION RATIONALE	SPECIAL SAMPLING AND ANALYTICAL REQUIREMENTS	ASTM	
Sulfide	SSMS	As for arsenate.	As for arsenate.		Titri-metric	Method provides a rapid and accurate technique for measurement of sulfide.	Sample is acidified and stripped with an inert gas and collected in a zinc acetate solution. Iodine solution is added to collection vessels, acidified with HCl and back titrated with 0.025 N sodium thiosulfate solution.	00745	Manual for Chemical Analysis of Water and Waste Water
Sulfite	Color-imetric	Method provides a rapid, simple analysis technique.	As specified by reagent test kit.		Titri-metric	Method provides a rapid and accurate technique for measurement of sulfite. ASTM method.	Air is excluded while sample is being taken by use of apparatus described in reference. HCl, KI and KIO_3 are added. Excess iodine chloride formed is titrated with 0.01 N $Na_2S_2O_3$ using a dead stop endpoint - indicating apparatus.	D1139	Starch indicator may be used.
Sulfate	Turbid-imetric/ Color-imetric	Method provides a rapid, simple analysis technique.	As specified by reagent test kit.		Gravi-metric	Method provides an accurate technique for measurement of sulfate. ASTM method.	Sample is filtered, pH adjusted to 4.5 with HCl, hot $BaCl_2$ added, allowed to stand for 2 hours, filtered and ignited at 800°C.	D516	A titrimetric method may also be used with $BaCl_2$, titrating in an alcoholic solution to a thorin endpoint.

4. Individual particle anion composition
5. Individual particle solubilities
6. Weight loss with respect to temperature
7. Bulk elemental distribution

Having completed the initial elemental and anion screening tests, primary compound identification methodology can now be applied.

5.2.2 Bulk Composition Characterization

It is expected that the samples will have to be dried to a constant water content to improve both IR and XRD spectra. Information from the TGA/DSC step will be used to select a drying temperature that provides water removal without sample decomposition. Further sample preparation will vary with the requirements of the specific analysis method.

For IR analysis, the KBr pellet technique for qualitative analysis is not recommended due to ion exchange possible during the pelletizing process. It is recommended that a Nujol mull of the sample and AgCl (1333-400 cm^{-1}) and polyethylene (600-45 cm^{-1}) windows be used. Interpretation of the infrared spectra on the basis of characteristic frequencies can provide the identity of specific anions and some individual compounds. General absorption regions for several anions are given in Table 5-2, and specific absorption bands, which have been observed in particulate samples, are listed in Table 5-3.

Several investigators have done extensive work with inorganic compounds and have been able to produce specific correlations between observed spectra and individual compounds. Tables 5-4 and 5-5 list the characteristic bands for several nitrates and sulfates which could be present in environmental samples. There are definite analytical frequencies which can be used to identify compounds, particularly when supporting elemental analysis information is available.

Electron spectroscopy for chemical analysis (ESCA) will be performed on both loose particulates and particles collected on filters. Loose

Table 5-2. Useful IR Bands

Anion	Absorption Bands (cm^{-1})
$SO_4^=$	610 - 690 (m)
	1080 - 1130 (s)
NO_3^-	610 - 640 (m, sp)
	1350 - 1370 (s)
$CO_3^=$	650 - 680 (m)
	1430 - 1450 (s)
$SiO_3^=$	∿900 - 1100 (vs)

particulate samples can be attached to a sample holder using an approach called the "sticky gold" technique. This technique was devised to overcome the conductivity problem and securely mount the sample. It sandwiches the sample between two layers of sputtered gold. The first layer applied to the scotch tape does not change the tackiness of tape, which allows loose particles to be stuck to the surface. Another layer of gold is deposited to assure that all the particles are near a conductive surface. Filter pieces can be clamped directly onto the sample holder after the the bottom layers of the filter have been peeled off.

Table 5-3. Listing of Assigned Infrared Bands Observed in Particulate Samples

Frequency, cm^{-1}	Species
3140	NH_4^+
3020	NH_4^+
2920	HYDROCARBON (C-H)
2860	HYDROCARBON (C-H)
2800	NH_4^+
1768	NO_2^-(BULK)
1720	NH_4^+ (HALIDE)
1620	H_2O
1435	CO_3^{2-}
1400	NH_4^+
1384	NO_3^- (SURFACE)
1360	NO_3^- (BULK)
1190	$PO_4^{3-(2)}$
1140	$PO_4^{3-(2)}$

Table 5-3. Listing of Assigned Infrared Bands Observed in Particulate Samples (Continued)

Frequency, cm^{-1}	Species
1120	PO_4^{3-}(2)
1110	SO_4^{2-}
1035	SiO_4^{4-}
980	SO_4^{2-}
880	CO_3^{2-}
840	NO_3^-(BULK)
800	SiO_4^{4-}
780	SiO_4^{4-}
728	CO_3^{2-}
670	PO_4^{3-}
627	PO_4^{3-}
620	SO_4^{2-}
600	PO_4^{3-}
470	SiO_4^{4-}

Table 5-4. Infrared Bands of Some Common Nitrates (cm^{-1})

Compound	Band Category(a)				
	VW	W	M	S	VS
$NaNO_3$	2428		836 sp		1358
					1790
KNO_3			824 sp		1380
					1767
$Ca(NO_3)_2 \cdot XH_2O$	1044		820 sp	~1430	~1350
				~1640	
$Fe(NO_3)_3 \cdot 9H_2O$			835 sp	1615	1361
					~1785
$Ca(NO_3)_2 \cdot 3H_2O$	2431		836 sp	1587	1378
					1790
$Pb(NO_3)_2$		807	726		1373
			836 sp		

W = Weak, M = Medium, S = Strong, V = Very, SP = Sharp, B = Broad

Table 5-5. Infrared Bands of Some Common Sulfates (cm^{-1})

Compound	Band Category^(a)				
	VW	W	M	S	VS
Na_2SO_4		645		620	1110
K_2SO_4					1110 620
$CaSO_4 \cdot 2H_2O$		1010 (sh) 1670	318 2200 (b)	603 667 1630 (sp) 3410 (b)	1130 (vb)
$MnSO_4 \cdot 2H_2O$		510 (vb) 607	660 1025	825 3225 (b)	1135 (vb)
$FeSO_4 \cdot 7H_2O$	990	1150 (sh)	1625	611 (vb) 3330 (b)	1090 (vb)
$CuSO_4$		1020 (sp) 1600 (sh)	680 805 860	1200 ∿3300 (b)	1090 (vb)
$PbSO_4$			592 (sp) 623 (sp)		

(a) V = Very, W = Weak, M = Medium, S = strong, SH = shoulder, B = Broad, SP = sharp

ESCA is employed in this step of the analysis to provide information on the oxidation state of the elements present on the surface of the sample, as well as on the change in elemental composition as various monolayers of sample material are removed through ion etching.

Knowing the oxidation state of elements such as Cl, S, V, Mo, and Si provides indications to the presence or absence of specific compounds. Once ESCA has qualitatively established the presence of a species, specific quantitative wet chemical tests can be made. In some cases the chemical shift information has been correlated with specific compounds.(10) In this case direct determination of a compound is possible. It should be noted that ESCA is limited to bulk concentrations of 0.1 percent or more. However, it is an extremely sensitive surface technique which is capable of seeing a monolayer of a given element. Ar^+ etching can be used to verify the homogeneous composition of the sample, or to perform elemental depth profile analysis.

The performance of specific anion tests, IR analysis, and ESCA establishes substantial information on the concentrations of a variety of cations and anions in the sample. This information simplifies interpretation of the XRD spectra, and provides an independent quantification of the species present. In X-ray diffraction (XRD) analysis, approximately 100 mg of material are ground in an agate mortar, ultrasonically dispersed with a 1:4 mixture of collodion with alcohol and then evenly spread over a glass support. Mounting in this fashion will produce the highest sensitivity at low 2θ values. The major disadvantage of XRD as an analytical tool is its inability to detect noncrystalline materials. In many environmental samples, the crystal structure of a compound could be grossly affected by the conditions at the source or those during sampling. For example, As_2O_3 can be amorphous or crystalline depending on its temperature history. Furthermore, the sensitivity of XRD is normally limited to 1 percent or higher although new computer averaging techniques enable materials to be detected in concentrations as low as 0.05 percent.

Having completed these analyses, information will have been obtained on the following:

1. Anions present
2. Valence state of elements present
3. Elemental depth profile
4. Major compounds present

At this point the analyst must correlate all data and determine if a reasonable (based on the analyst's judgment) agreement has been reached with the MEG elements exceeding their MATE values. If there is reasonable agreement between the elemental data obtained from quantitative techniques and the compounds determined in this characterization, further work should be carefully evaluated in terms of potential needs and end use.

5.2.3 Individual Particle Characterization

It should be emphasized that this phase of the analysis should be carried out at the analyst's discretion. The analyst should consider the sample, its source, the information already available, the type of information which is lacking, the instrumental techniques available, and analysis cost before proceeding.

Analytical techniques which are suggested for identification of individual particles include Scanning Electron Microscopy with Energy Dispersive X-ray spectrometry (SEM-EDX), Electron Probe Microanalysis (EPMA) and Transmission Electron Microscopy with Selected Area Electron Diffraction (TEM-SAED).

In SEM the sample specimen is swept by an electron beam and the variation of the secondary electron emission intensity is recorded. This signal simultaneously modulates the brightness of an oscilloscope beam, producing an image of the sample surface on the oscilloscope screen. Since the secondary electron beam is localized in the area impacted by the incident radiation, images of relatively high resolution are achieved which can provide morphological characteristics of individual particles. When SEM is used in conjunction with an energy dispersive X-ray spectrometer (EDX), the secondary X-rays produced can be monitored, thereby allowing identification and quantification of individual elements present in the sample. Determining the elemental distribution of a particle is particularly useful for those particles composed of various occluded materials; the high resolution and magnification of the SEM can produce images distinctive enough to identify the particle. As such, the SEM information is a valuable adjunct to the PLM, especially for particles smaller than 0.5μ.

In order to reduce the mounting time for both SEM and EPMA (electron probe microanalysis), particles should be mounted on a gold stage to provide a conductive surface. Normally, a carbon film would be deposited on the sample to ensure its conductivity. If the sample is reasonably conductive and long analysis times are not necessary, then the carbon film may be omitted. Mounting samples in this fashion will not interfere with later EPMA analysis.

Disadvantages of the method include the inability of SEM-EDX to detect the elements in the periodic table between carbon and sodium. Also, resolution of the elements between sulfur and nickel is limited. Although relatively long counting times are required for elements present in trace amounts, the stability of the EDX instrument limits counting time to 10 to 15 minutes. Peak broadening, interference from neighboring particles and difficulties in obtaining suitable matching standards can also limit the certainty of an analysis. The result is generally a bulk composition correlated with each particle type present in the sample matrix.

In EPMA a small energetic electron beam impinges the surface of the particulate specimen and produces characteristic X-ray emissions. EPMA can be used to qualitatively and quantitatively determine the elemental composition of particles ranging in size from 20μ down to about 0.2μ for most of the elements of atomic numbers above that of carbon. Instruments using wavelength dispersive X-ray spectrometers can resolve spectra of elements sulfur through nickel in atomic number. Using this detection, qualitative analyses are possible. Peak heights, or intensity ratios, are measured on samples and standards to provide a quantitative analysis.

To achieve the best accuracy, it is necessary to do a considerable amount of sample preparation. In most cases it is necessary to have standards similar in particle size and composition to the sample being analyzed. Further, identification is possible only for particles containing discrete compounds rather than a homogeneous mixture.

Transmission Electron Microscopy with Selective Area Diffraction (TEM-SAED) also involves the impingement of an electron beam on a thin film (~1500Å) of sample. The resulting single particle X-ray diffraction pattern permits identification of crystalline compounds. The qualitative and quantitative data obtained are excellent because 1) individual particles and fibers can be observed and identified, and 2) the use of selected area electron diffraction is a dependable technique for identification of such chemical species as asbestos and silica.

Combining the information derived from TEM-SAED and EPMA can aid the analyst in assembling the total nature of the various species present. Many substances which appear essentially identical in elemental composition as measured with the electron probe, will be determined by TEM-SAED to have a unique morphology and, therefore, their emitted nature and source clearly indicated.

At this point, if all the compounds for MEG elements exceeding their MATE values have not been found, then the analyst might choose to reduce the sample matrix into simple mixtures. He can either run magnetic density gradient, or selective dissolution studies. In magnetic separation, magnets are used to remove the magnetic fraction from the sample. In density gradient separation, particles are floated in organic solvents of known density. Considerable care must be used in selecting solvents because many compounds could be soluble in the solvents. This procedure can be used to obtain gross separations by density or can be used to determine individual particle densities. Selective dissolution uses a variety of solvents to remove more and more of the sample and in the process simplifying the composition of the residue.

In all these techniques care must be taken to avoid contamination and scrambling of compounds. Also, reasonably large quantities of sample are necessary. The end result of these separations is to provide less complex fractions which can be studied starting at the bulk characterization level.

6. LEVEL 2 ANALYSIS OF RETAINED SASS SAMPLES FOR ORGANIC COMPOUNDS

This Level 2 organic analysis plan is based on Level 1 analysis data and is intended for use on retained SASS samples. The plan assumes that Level 1 analysis has been completed and that this information is available. The techniques discussed should be implemented by a skilled mass spectrometrist, since at several points in the analysis, judgement and even modifications may have to be made to the procedures, depending on sample source or what compounds are identified during the course of the analysis.

Combined gas chromatography and mass spectrometry (GC/MS) is central to this analysis plan. GC/MS combines the separation power of the gas chromatograph with the unexcelled identification potential of the mass spectrometer. The incorporation of a computer based data handling system with the GC/MS provides the most powerful compound identification technique available to the analyst. The technique is cost effective but requires an experienced spectrometrist to suitably apply it to environmental samples and analyze the data generated. Judgements as to sample size, depending on instrument sensitivity, and mass range to be scanned, depending on instrument resolution, as well as selection of an alternate GC column for a specific sample, are at the discretion of the analyst. General direction is given in this report; however, a total analysis to identify every compound present in a complex mixture requires on-the-spot modification of procedures. The molecular weights and necessary M/e values for most of the MEG organic compounds are given in Appendix C.

The most cost effective Level 2 analysis scheme would be a specific analysis based on category data obtained from Level 1. This information would provide data for GC column selection and would generally simplify the overall analysis. The analysis scheme as outlined is for all categories of compounds on the MEG list with the exception of those compounds which are volatile and are analyzed by the field GC technique and those which are reactive and chemically modified by sampling or storage.

This Level 2 analysis plan incorporates wet chemical separations, including sample extractions and liquid chromatography, and instrumental analysis using primarily GC/MS. Other techniques are discussed which may be applied in special cases but require further research into their application. These include high resolution mass spectrometry (HRMS), chemical ionization mass spectrometry (CIMS), gas chromatography, with selective detectors, and capillary column GC/MS. The proposed analysis plan is patterned after Level 1. It was designed to provide information on total compound identity and yet ease the total sample burden imposed by the analysis of every Level 1 organic sample fraction. Specific GC columns are described together with appropriate conditions for their use in identifying the appropriate MEG compounds sought in each fractionated sample extract. If a category is known to be absent in a specific sample, based on information from Level 1, it is expected that this knowledge will be used to modify the analysis. If specific compounds are expected at very low concentrations, they should be analyzed separately since, in general, low levels of materials will be lost in the analysis plan as outlined. Typical sensitivites for various analysis steps are given as a part of the overall method discussion. It is important that the analyst implementing this Level 2 plan have a working knowledge of Level 1 organic analysis since it is not intended to be a step-by-step workbook but rather a logical sequence of experiments to achieve the goal of compound identification.

The approach presented here has been specifically developed for Task 6 of EPA Contract 68-02-2163. Under EPA Contract 68-02-2150 a procedures manual is being prepared for Level 2 organic sampling and analysis (5). It should be referred to by the Level 2 analyst as a more complete compendium on organic compound identification methodologies.

6.1 HARDWARE REQUIREMENTS AND OPTIONS FOR LEVEL 2 ANALYSIS

The primary tool for Level 2 analysis is a high sensitivity GC/MS instrument. A discussion of corollary GC/MS techniques, expected to be useful during the course of this analysis, is given as well as where their use is appropriate. Other instrumental methods are briefly discussed, however, further work is needed before they may be routinely applied.

6.1.1 GC/MS

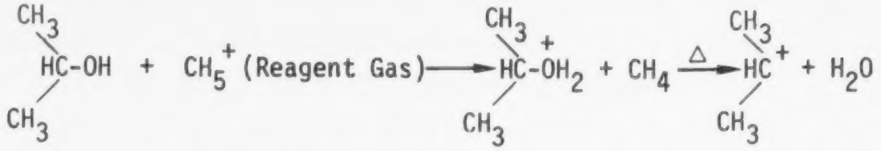
In order to apply GC/MS and obtain reliable data it is necessary to have a spectrometer which is capable of high speed scanning (i.e., recording a full mass spectrum in 3 seconds or less) with resolution that will allow separation nominal mass peaks to at least mass 600. The gas chromatograph should be capable of using glass columns since many of the materials to be analyzed are sensitive to metal surfaces. The mass spectrometer should be capable of chemical ionization with a variety of reagent gases such as methane or isobutane. The ability to use capillary columns may be useful in many of the analyses which are anticipated. The incorporation of a computer based data handling system lessens the labor involved in acquiring mass spectral data, and reduces the time for data reduction and interpretation. The computer does not eliminate the need for an experienced mass spectroscopist; it merely provides a more cost effective means of handling large columns of mass spectral data.

6.1.2 Chemical Ionization (CI) Mass Spectrometry

Normal mass spectrometry is accomplished by bombarding the sample with 70 eV electrons. The ionization process produces a spectrum which contains characteristic fragment ions from the molecule under study. In most cases a molecular ion is produced (i.e., the ion representative of molecular weight) and its identification is unambiguous; however, in some cases, no molecular ion is produced or it is present at such a low level that it cannot be identified. The most important peak in any mass spectrum is the molecular ion since a knowledge of molecular weight reduces the total number of organic compound possibilities by a substantial amount. Electron ionization does provide a great deal of compound structure information, but when the molecular ion is absent much information is lost, making spectral interpretation difficult.

Chemical ionization incorporates a reagent gas to perform the ionization process. The use of methane or isobutane for the chemical ionization process is most common. When these reagent gases are used, the energy of ionization is reduced from 70 to about 7 eV. The result is ionization of a sample without sufficient excess energy to cause significant fragmentation and, in most cases, the pseudomolecular ion dominates the spectrum yielding molecular weight information. The CI process involves a transfer of a proton from the reagent gas to the sample when ionization occurs. The resulting spectrum is a pseudomolecular ion at 1 mass unit higher than the molecular weight of the compound. Chemical ionization should always be used in conjunction with electron ionization for spectral interpretation.

As is true with most analytical techniques, chemical ionization is not without its difficulties. The ionization of some materials such as alcohols, causes a protonation of the hydroxyl group followed by a loss of water from the pseudomolecular ion by a thermal process. An example of this type of ionization is given below:



The loss of water from the pseudomolecular ion is primarily dependent on source temperature, and is increased with higher temperatures. This fragmentation process may not take place when electron ionization is used and in many cases causes confusion in the interpretation of the molecular weight. Similar occurrences take place when amines are being studied, showing a loss of ammnonia from the pseudomolecular ion, and to a lesser extent acids, ethers, esters, and halogenated compounds. Hydrocarbon samples are typically not sensitive to chemical ionization. This is especially true of normal hydrocarbons. Under CI conditions, straight chain hydrocarbons often show a loss of 1 from the molecular ion rather than an addition, together with a significant reduction in overall sensitivity. Materials which contain heteroatoms such as nitrogen, oxygen, and sulfur, generally show an increase in sensitivity relative to their electron ionization spectra. This variation in sensitivity is useful in identifying heteroatom structures in complex hydrocarbon samples using chemical ionization. Other reagent gases are available (e.g., ammonia, nitrous oxide, and hydrogen), however, less work has been done with these reagent gases and their use should be limited to those experienced in their application.

6.1.3 Multiple Ion Detection Mass Spectrometry

Multiple ion detection mass spectrometry (MID) is a technique to improve the sensitivity and selectivity of the mass spectrometer as a GC detector. To use MID the GC retention time of the compound of interest must be known in advance and this, together with the appearance of a peak at that retention time, is used as confirmation of the presence of a material. To use the technique, the mass spectrometer is set up to monitor a specific set of masses, typically a number from 1 to 8. The sensitivity of the mass spectrometer is increased by increasing the amount of time spent on selected mass peaks. The signal to noise is improved as a square root of 2 for every doubling of the time spent at a given mass number. This specifically is achieved by selecting only a small number of mass peaks to be monitored specifically for the compound of interst. The resultant data is a chromatogram for a specific set of masses. When an inflection occurs at a point consistent with the retention time of the compound of interest, that material has been identified and can be quantified providing standards are available. It is not practical to perform multiple ion detection for several components in a single mixture, however, if a particular compound such as a nitrosoamine is suspected to be present in a sample at very low levels, the technique can be invaluable. Standards should be run to determine the retention time on the column used for the analysis. Any interferences should also be noted.

6.1.4 High Resolution Mass Spectrometry (HRMS)

The techniques discussed to this point require that the compound of interest be amenable to gas chromatography. Many materials, of course, can not be chromatographed and therefore do not lend themselves to GC/MS. High resolution mass spectrometry is a technique by which one can analyze low volatility residual materials. Total compound identification may not be possible in all cases depending on mixture complexity; however, functional groups and heteroatoms can generally be identified unambiguously. The technique as discussed employs the direct insertion probe which is used to introduce the sample into the ion source of the mass spectrometer. The use of a high resolution data system, together with the high resolution mass spectrometer, is important in obtaining useful data in a reasonable time. Full spectra should be recorded and the computer used to reduce the data to element maps for selected mass peaks. The element maps will give the elemental composition for mass peaks and an experienced mass spectrometrist can use this information to determine the compound types in the sample. The sophistication of a high resolution mass spectrometer is much greater than GC/MS and the sophistication of the operator must also be greater.

This technique should be applied when it is evident that significant quantities of organic compounds have not been chromatographed and, therefore, are unidentified through the application of GC/MS.

6.1.5 Infrared Spectroscopy

Infrared spectroscopy is useful in determining compound functionality. The technique is not applied directly in this Level 2 plan since it is assumed that IR spectra have previously been recorded on all liquid chromatography fractions in Level 1. It is also assumed that this information is available and is used by the analyst to select appropriate GC columns and to ensure that he has analyzed all materials which are present in Level 1 by this Level 2 plan.

6.1.6 High Pressure Liquid Chromatography (High Resolution Liquid Chromatography, HPLC)

High pressure liquid chromatography is not discussed in detail in this analysis plan. This is not to say the technique is not useful; in fact, it may be the most important technique to be ultimately used for Level 2 analysis. HPLC does not suffer from the need for volatility of a sample as is true with gas chromatography. It is a very powerful separation tool, superior to the extraction techniques which are used in this plan to grossly separate organic compounds. It may be possible through research to use HPLC as a screening tool to provide compounds class information.

Further work must be done before the technique can be universally applied to a Level 2 analysis scheme. In the future, it may be possible to take fractions from a high resolution liquid chromatograph for direct probe analysis and mass spectrometer identification or ultimately an interface between the liquid chromatograph and the mass spectrometer in much the same way as a gas chromatogaph. Until further work is done using HPLC on Level 2 type samples, it remains a highly probable technique rather than a highly useful one.

6.1.7 Capillary GC/MS

It may be found in many cases, that packed columns cannot provide the chromagraphic resolution necessary to obtain good mass spectral data. It is expected that this will be especially true in the direct analysis of the extracts prior to concentration or liquid chromatographic fractionation.

A wide variety of capillary columns are available, including standard open tubular wall coated columns, SCOT columns, and micropacked columns. For the beginner, the use of a SCOT column is recommended since it is more tolerant of temperature and sample size while providing increased resolution over its packed column counterpart. The liquid phase chosen for a capillary column is generally based on information obtained using packed columns. A wide variety of liquid phases are available, however, due to their expense, only a selected few columns are expected to be used routinely. For general application in Level 2 analysis, it is recommended that a laboratory have available an OV - 17 SCOT column and a Carbowax 20M SCOT column, which are between 50 and 100 feet in length. These two columns will satisfy 90 percent of the requirements for capillary column GC.

6.2 SAMPLE PREPARATION AND EXTRACTION PROCEDURES

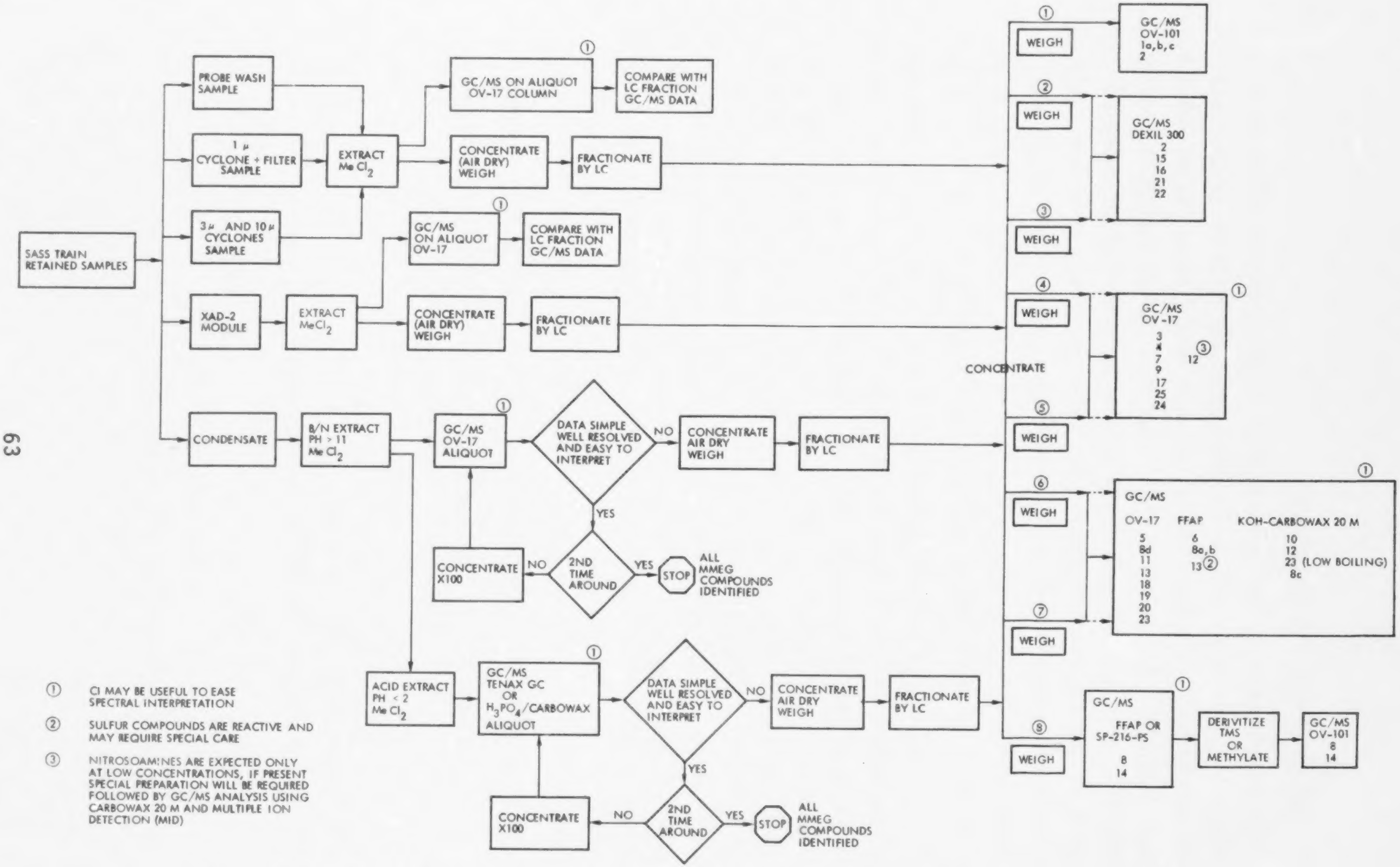
The preparation and extraction procedures described in this section are very similar to those used in the Level 1 analysis plan. For those analysts familiar with Level 1 analysis, the only modification is in the extraction of the condensate of the XAD-2 sorbent trap. Level 1 prepared samples should be used where possible without further work.

6.2.1 Probe Wash, Cyclones, and Filter SASS Train Samples

The probe wash, cyclones, and filter samples should be analyzed as shown in Figure 6-1 starting with a methylene chloride extraction. The extractions should be made using a Soxhlet apparatus for 24 hours. For particulate samples, the Soxhlet cup should have been previously extracted following the established procedures outlined for Level 1 analysis to remove contamination which would lead to erroneous results.

6.2.2 XAD-2 Sorbent Trap

The XAD-2 resin from the sorbent trap should be extracted with methylene chloride using a glass cup and a large Soxhlet apparatus.


SASS TRAIN RETAINED SAMPLES
PROBE WASH SAMPLE
1 μ CYCLONE + FILTER SAMPLE
3 μ AND 10 μ CYCLONES SAMPLE
XAD-2 MODULE
CONDENSATE
EXTRACT $MeCl_2$
GC/MS ON ALIQUOT OV-17 COLUMN ①
COMPARE WITH LC FRACTION GC/MS DATA
CONCENTRATE (AIR DRY) WEIGH
FRACTIONATE BY LC
EXTRACT $MeCl_2$
GC/MS ON ALIQUOT OV-17 ①
COMPARE WITH LC FRACTION GC/MS DATA
CONCENTRATE (AIR DRY) WEIGH
FRACTIONATE BY LC
B/N EXTRACT PH > 11 $MeCl_2$
GC/MS OV-17 ALIQUOT ①
DATA SIMPLE WELL RESOLVED AND EASY TO INTERPRET
NO
YES
CONCENTRATE AIR DRY WEIGH
FRACTIONATE BY LC
2ND TIME AROUND
NO
YES
CONCENTRATE X100
STOP
ALL MMEG COMPOUNDS IDENTIFIED
ACID EXTRACT PH < 2 $MeCl_2$
GC/MS TENAX GC OR H_3PO_4/CARBOWAX ALIQUOT ①
DATA SIMPLE WELL RESOLVED AND EASY TO INTERPRET
NO
YES
CONCENTRATE AIR DRY WEIGH
FRACTIONATE BY LC
2ND TIME AROUND
NO
YES
CONCENTRATE X100
STOP
ALL MMEG COMPOUNDS IDENTIFIED
CONCENTRATE
① WEIGH
② WEIGH
③ WEIGH
④ WEIGH
⑤ WEIGH
⑥ WEIGH
⑦ WEIGH
⑧ WEIGH
GC/MS OV-101 1a,b,c 2
GC/MS DEXIL 300 2 15 16 21 22
GC/MS OV-17 3 4 7 9 17 25 24 12③ ①
GC/MS ①
OV-17 5 8d 11 13 18 19 20 23
FFAP 6 8a,b 13②
KOH-CARBOWAX 20 M 10 12 23 (LOW BOILING) 8c
GC/MS FFAP OR SP-216-PS 8 14 ①
DERIVITIZE TMS OR METHYLATE
GC/MS OV-101 8 14

① CI MAY BE USEFUL TO EASE SPECTRAL INTERPRETATION

② SULFUR COMPOUNDS ARE REACTIVE AND MAY REQUIRE SPECIAL CARE

③ NITROSOAMINES ARE EXPECTED ONLY AT LOW CONCENTRATIONS, IF PRESENT SPECIAL PREPARATION WILL BE REQUIRED FOLLOWED BY GC/MS ANALYSIS USING CARBOWAX 20 M AND MULTIPLE ION DETECTION (MID)

Figure 6-1. General Logic Flow Chart for Level 2 Organic SASS Component Analysis

6.2.3 Extraction of the Condensate

The condensate from the sorbent trap should first be extracted with methylene chloride after the pH has been adjusted to 11 or greater with 6N sodium hydroxide. This base/neutral extract should be set aside for subsequent analysis. The solution pH is then adjusted to less than 2 using 6N hydrochloric acid and the extraction with methylene chloride repeated. This division of the condensate sample into two extracts may eliminate the need for the liquid chromatographic (LC) separation step making the overall analysis less expensive.

6.3 ANALYSIS OF THE EXTRACTS FOR VOLATILE COMPONENTS

Concentration of extracts prior to analysis causes the loss of most materials with boiling points below about 220°C (C12). To obtain data on low boiling extracted compounds from the SASS train samples, GC/MS analysis is run on the sample prior to concentration. A 2 ml aliquot of the extract should be saved for this analysis. One GC/MS run on each sample is made using a general purpose column, e.g., OV - 17. If specific classes of compounds were found to be present from the Level 1 data for a given extract, a repeat analysis of the uncondensed extract may be necessary to determine if more volatile materials in the same compound class are present. Column selection for the rerun of a sample should be based on the categories identified from the GC/MS analysis of the LC fractions.

6.3.1 GC/MS Analysis of the Probe Wash, Cyclones, and Filter Extract

The GC/MS analysis of the probe wash, cyclones and filter extracts should be run using chromographic conditions given below:

- Liquid phase: OV-17
- Liquid loading: 3 percent
- Solid support: Chromasorb W - AW - DMCS
- Column type: glass
- Column size: 2 mm ID x 2 meters long
- Temperature program: 50°C for 5 minutes, 50°-280°C at 6°C per minute

- Hold at maximum until all peaks elute
- Injector temperature: 280°C
- Detector and transfer line to separator temperature: 280°C
- Flow rate of helium: 30 cc per minute
- Sample size: 1 to 5 μl
- Sensitivity: approximately 30 nanograms per μl injected

This set of chromatographic conditions is very general and is designed to separate and quantify most organic compounds suspected to be present in a sample. Specific categories and concentrations determined from the LC fractionation step may dictate the use of an alternate column and/or modification of the conditions for this column. This judgement can be made only by the analyst based on his ability to interpret the GC/MS data. The complexity of this extract is expected to vary widely depending on the source. When a sample is highly complex, the use of chemical ionization mass spectrometry is recommended, chemical ionization may aid in the interpretation of individual mass spectra especially if no molecular ion is present in the EI spectrum. The application of chemical ionization was described in Section 6.1.2.

When problems of chromatographic resolution are present due to sample complexity the use of capillary GC/MS may aid compound identification. Liquid phase selection should be made based on the LC fraction data. A good starting column would be a 50-foot OV-17 SCOT column. The application of capillary GC/MS is discussed in Section 6.1.7.

6.3.2 GC/MS Analysis of the XAD-2 Module Extract

The procedure outlined for the probe, cyclones, and filter extract, Section 6.3.1, is adequate for the methylene chloride extract of the XAD-2 sorbent material. No special precautions other than those discussed above are necessary. The sensitivity of this analysis is also expected to be 30 nanogram per μl injected.

6.3.3 GC/MS Analysis of the Condensate Extract

The condensate extract consists of two parts, a base/neutral fraction and an acid fraction. The GC/MS analysis of these fractions is based on the polarity of compounds expected to be present. The separation of the condensate into two parts may possibly eliminate the need for an LC fractionation step on this sample. The base/neutral fraction may be somewhat complex but the acid fraction should be relatively clean. If the chromatograms are not too complex, it is advisable to concentrate the samples hundredfold and repeat this analysis to increase the overall sensitivity without the necessity of LC fractionation. A probe HRMS run on the residue of the sample will provide information on the compounds which are not amenable to GC/MS (see Section 6.1.4 for high resolution mass spectrometry techniques). If both fractions are complex, the samples should be blended prior to LC fractionation, however, if only one fraction is complex, only that fraction need be submitted for further workup.

GC/MS Analysis of the Base/Neutral Fraction of the Condensate _ _

The same procedure outlined for the probe, cyclones, and filter extracts is applied to the base/neutral fraction of the condensate. Sensitivity of this analysis is expected to be 30 nanograms per microliter injected.

GC/MS Analysis of the Acid Fraction of the Condensate _ _

Due to the polarity and the acidic nature of the acid fraction a polar column is used for this analysis. The exact column to be used requires some additional information or the use of two columns for analysis. If phenols are expected, for instance, a Tenax GC column would be chosen. If carboxylic acids are expected, a phosphoric acid treated carbowax 20M column would be the best choice. The use of both Tenax and phosphoric acid treated carbowax will give results on all acidic species which could be present in the sample. When using the carbowax column, phosphoric acid treated glass wool should be used to plug the column ends. This will minimize adsorption of acidic species. The gas chromatographic procedure for each of the columns is given below:

- Column type: Tenax GC
- Column material: glass

- Column size: 2 mm ID x 2 meter long
- Temperature program: 50°C for 5 min, 50°-300°C at 6° min; hold at maximum until all peaks elute
- Injector temperature: 280°C
- Detector and transfer line to separator temperature: 280°C
- Flow rate of helium: 30 cc per minute
- Sample size: 1 to 5 μl
- Sensitivity: 100 nanograms per μl injected

Tenax GC is a gas-solid chromatographic material. It does not contain a liquid phase and has very good temperature stability. It tends to elute polar materials with ease, however, nonpolar compounds are likely to be retained on the column. The ultimate sensitivity achieved with this column is somewhat lower than many others due to its absorbtive character.

The following chromatographic conditions are for the phosphoric acid treated carbowax 20M column:

- Liquid phase: 3 percent phosphoric acid and 10 percent carbowax 20M
- Solid support: Chromasorb W-AW
- Column type: glass
- Column size: 2 mm ID x 2 meter long
- Temperature program: 50°C for 5 minutes; 50°-180° at 4°C per minute. Hold at maximum until all peaks elute
- Injector temperature: 190°C
- Detector and transfer line to separator temperature: 190°C
- Flow rate of helium: 30cc per minute
- Sample size: 1-5 μl
- Sensitivity: 50 nanograms per μl injected

Alternate column for acid fraction of condensate extract:

- Liquid phase: FFAP (Free Fatty Acid Phase)
- Liquid loading: 10 percent

- Solid support: Chromasorb W-AW
- Column type: glass
- Column size: 2 mm ID x 2 meter long
- Temperature program: $50^{o}C$ for 5 minutes, 50^{o}-230^{o} at 6^{o} per minute; hold at maximum until all peaks elute
- Injector temperature: $240^{o}C$
- Detector and transfer line to separator temperature: $240^{o}C$
- Flow rate of helium: 30cc per minute
- Sample size: 1-5 μl
- Sensitivity: variable with sample from 30 to 100 nanograms per μl injected

The conditions specified for the various columns may be modified to improve a specific analysis. When extracts are found to be relatively clean, a faster temperature program will result in less analysis time per sample. This judgement must be made by the operator at the time of analysis.

6.4 LIQUID CHROMATOGRAPHIC (LC) SEPARATION

Once the preliminary GC/MS work has been completed on the extracted samples, a general idea of compound type or class is available. The next step is to separate the various extracts after they have been condensed to identify specific compounds by GC/MS. The purpose of this LC procedure is to separate the samples into approximate classes based on polarity using a gradient elution LC technique. The detailed procedure for the LC fractionation is given in Appendix B. The LC separation is not a high resolution technique therefore overlap in the compound classes in many of the fractions is common. The procedure for Level 1 is followed even though several of the fractions are blended after separation prior to analysis. The blending of fractions is due to compound class similarity and allows a more cost effective GC/MS analysis. Table 6-1 gives the blending of the fractions following the LC separation using the solvent gradient outlined in Table 6-2. (Unblended aliquots can be analyzed if the analyst decides complexity warrants it.)

Table 6-1. LC Fraction Blending

LC Fraction	Blend
1	A
2	B
3	
4	C
5	
6	D
7	
8	E

Table 6-2. Solvents Used in Liquid Chromatographic Separations

Fraction No.	Solvent Composition
1	Pentane
2	20% Methylene Chloride in Pentane
3	50% Methylene Chloride in Pentane
4	Methylene Chloride
5	5% Methanol in Methylene Chloride
6	20% Methanol in Methylene Chloride
7	50% Methanol in Methylene Chloride
8	5/70/30, Conc. HCl/Methanol/Methylene Chloride

6.5 GC/MS ANALYSIS OF LC FRACTIONS

Each of the blended LC fractions are concentrated to a volume of less than 10 ml using an air drying technique. Once this has been achieved, 1 ml of the internal standard is added and diluted to exactly 10 ml using methylene chloride in a volumetric flask. In specific cases, where sensitivity is very important, a smaller volume may be used as long as it is known exactly. The GC analysis of the individual fraction blends is discussed below.

6.5.1 Fraction A (1)

Fraction A contains the compounds that generally fall in categories 1 and 2 of the MEG list (see Table 2-5). These include aliphatic hydrocarbons and alkyl halides. These are the least polar compounds to be analyzed and are well-suited to low polarity silicon liquid phase GC columns. The column conditions given below provide complete analysis of this fraction.

- Liquid phase: OV-101
- Liquid loading: 3 percent
- Solid support: Chromasorb W-AW-DMCS
- Column type: glass
- Column size: 2 mm x 2 meters long
- Temperature program: 50^{o}C for 5 minutes, 50^{o}-280^{o} at 6^{o} per minute. Hold at maximum until all peaks elute
- Injector temperature: 290^{o}C
- Detector and transfer line to separator temperature: 290^{o}C
- Flow rate of helium: 30 cc per minute
- Sample size: 1-5 μl
- Sensitivity: 30 to 50 nanograms per μl injected

Because of the nature of the compounds found in fraction A, the use of chemical ionization is not recommended to improve sample identification. The loss in sensitivity and the confusion resulting from a mixed ionization process suggests that electron ionization is the method of choice.

6.5.2 Fraction B (2 and 3)

Fraction B has been blended and contains categories 2, 15, 16, 21, and 22 as outlined in Figure 6-1. These compounds can normally be classified as unsaturated hydrocarbons and halogenated species. In general, these classes produce strong molecular ions in the electron ionization mode of operation resulting in spectra which are easy to interpret.

The GC separation is best conducted on a high temperature nonpolar chromatographic column such as Dexil 300. The conditions for a typical analysis are given below.

- Liquid phase: Dexil 300
- Liquid loading: 3 percent
- Solid support: Chromasorb W-AW-DMCS
- Column type: glass
- Column size: 2 mm ID x 2 meters long
- Temperature program: 50^oC for 5 minutes, 50^o-300^oC at 6^o per minute. Hold at maximum until all peaks elute
- Injector temperature: 300^oC
- Detector and transfer line to separator temperature: 290^oC
- Flow rate of helium: 30 cc per minute
- Sample size: 1-5 μl
- Sensitivity: 10-30 nanograms per μl injected

The compounds generally found in LC fractions 2 and 3 are also not amenable to chemical ionization, and electron ionization spectra of these materials should be sufficient for compound identification.

If the mixture is exceedingly complex such that chromatographic resolution is insufficient, the use of a silicon liquid phase OV-17 capillary column is recommended.

6.5.3 GC/MS Analysis Fraction C (4 and 5)

Fraction C represents classes of compounds with increased polarity over the previous fractions. Several intermediate polarity nitrogen, sulfur, and oxygen containing compounds elute in these fractions. Analysis of this material is best suited to an intermediate polarity silicon column of which any one of several can be chosen. The chromatographic conditions given below represent a compromise for this class of materials.

- Liquid phase: OV-17
- Liquid loading: 3 percent
- Solid support: Chromasorb W
- Column type: glass
- Column size: 2 mm ID x 2 meters long
- Temperature program: $50^{o}C$ for 5 minutes; 50^{o}-$290^{o}C$ at $6^{o}C$ per minute. Hold at maximum until all peaks elute
- Injector temperature: $290^{o}C$
- Detector and transfer lines to separator temperature: $290^{o}C$
- Flow rate of helium: 30 cc per minute
- Sample size: 1 to 5 μl
- Sensitivity: 20 to 50 nanograms per microliter injected

Due to the nature of these classes of compounds and the fact that they generally contain heteroatoms, chemical ionization is recommended as a supplemental technique to aid in the interpretation of the mass spectral data.

6.5.4 GC/MS Analysis of Fraction D (6 and 7)

LC fractions 6 and 7 represent complex mixtures of compounds which are rather polar in nature and have widely varying acidities. In these two fractions both basic and acidic compounds elute together, and such mixtures are not amenable to a single gas chromatographic column. Without previous information as to the nature of compounds present, it is necessary to run this fraction on at least three different GC columns in order to ensure that all materials in the sample have been identified.

The columns selected for these analyses, given below, include an intermediate polarity silicon column, a column designed to elute free fatty acids and glycols and another to elute free amines. A class of compounds known as nitrosoamine elute in this fraction. These materials are very toxic even at low concentrations. An attempt to analyze for nitrosoamines in this mixture, without special care would be virtually impossible. If nitrosoamines are expected, special precautions should be taken; specifically designed cleanup steps should be used followed by chromatographic analysis with a column such as carbowax 20M which is especially good for nitrosoamines at low concentration. The use of multiple ion detection mass spectrometry for the determination of nitrosoamines would be logical for this type of sample. The three columns used for the analysis of fractions 6 and 7 are given below together with chromographic conditions:

- Liquid phase: OV-17
- Liquid loading: 3 percent
- Solid support: Chromasorb W
- Column type: glass
- Column size: 2 mm ID x 2 meters long
- Temperature program: $50^{\circ}C$ for 5 minutes, 50°-$300^{\circ}C$ at 6° per minute. Hold at temperature maximum until all peaks elute
- Injector temperature: $290^{\circ}C$
- Detector and transfer line to separator temperature: $290^{\circ}C$
- Flow rate of helium: 30cc per minute
- Sample size: 1 to 5 microliters
- Sensitivity: 20 to 50 nanograms per microliter injected

This column is designed to elute those compounds with intermediate polarity such as esters, ketones, and nitrogen heterocycles. The more polar materials are better suited to an FFAP column described below.

- Liquid phase: FFAP (Free Fatty Acid Phase)
- Liquid loading: 10 percent

- Solid support: Chromasorb W-AW
- Column type: glass
- Column size: 2 mm ID x 2 meters long
- Temperature program: $50^{0}C$ for 5 minutes; 50^{0}-$230^{0}C$ at 6^{0} per minute. Hold at temperature maximum until all peaks elute
- Injector temperature: $240^{0}C$
- Detector and transfer line to separator temperature: $250^{0}C$
- Flow rate of helium: 30 cc per minute
- Sample size: 1 to 5 μl
- Sensitivity: 50 to 100 nanograms per microliter injected

The basic compounds, such as amines, are better suited to columns specific for basic materials. The following set of conditions will provide good chromatographic separations for basic compounds.

- Liquid phase: 10 percent carbowax 20M-3 percent KOH
- Solid support: Chromasorb W
- Column type: glass
- Column size: 2 mm ID by 2 meters long
- Temperature programs: $50^{0}C$ for 5 minutes 50^{0}-$180^{0}C$ at $6^{0}C$ per minute. Hold at temperature maximum until all peaks elute
- Injector temperature: $180^{0}C$
- Detector and transfer line to separator temperature: $190^{0}C$
- Flow rate of helium: 30 cc per minute
- Sample size: 1 to 5 μl
- Sensitivity: 50 to 100 nanograms per microliter injected

The use of these three columns should provide compound identification on fractions 6 and 7. Alternate columns may be used if information from the GC/MS analysis of the original extracted material shows specific categories present. One alternate column would be Tenax GC, which is especially suited for analysis of glycols and amides, while Chromasorb 103 can be used as a substitute for the amine column KOH-carbowax.

6.5.5 GC/MS Analysis of Fraction E (8)

LC fraction 8 contains the most polar materials in the sample such as carboxylic acids and sulfonic acids. These compounds are not particularly well-suited to chromatographic analysis. Specific columns such as FFAP or phosphoric acid treated carbowax (SP-216-PS) can be used to run the lower boiling acidic species. However, the high boiling materials can only be analyzed by direct insertion probe mass spectrometry, preferably using high resolution. The GC analysis conditions using FFAP have previously been given and conditions for the other acid column are given below.

- Liquid phase: 10 percent Carbowax 20M-3 percent H_3PO_4
- Solid support: Chromasorb W AW
- Column type: glass
- Column size: 2 mm ID x 2 M long
- Temperature program: 50^oC for 5 minutes; 50^o-180^oC at 6^oC/min. Hold at temperature maximum until all peaks elute
- Injector temperature: 190^oC
- Detector and transfer line to separator temperature: 190^oC
- Flow rate of helium: 30cc/min
- Sample size: 1-5 μl

Acid fraction 8 is particularly well-suited for chemical ionization and, if high resolution mass spectrometer is not available for running the solids probe on the residue, a solids probe analysis using chemical ionization may aid in identification of some components.

An alternative to direct analysis of acidic species is derivation. Several methods of derivation are available. However, the two most common are the formation of trimethylsilyl esters of the acids or methylation to form the methyl esters. The trimethylsilyl esters are the easiest to form, although GC/MS identification of these materials can be quite difficult. GC/MS analysis of this derivative generally results in the TMS fraction of the molecule dominating fragmentation such that the spectrum is a function of the derivation rather than the molecule itself. The result is a confusing, uninformative spectrum that is difficult to interpret.

Chemical ionization is not particularly well-suited to any TMS derivative.

The formation of methyl esters of carboxylic acids and sulfonic acids is a more tedious process requiring considerably more chemistry and care. The derivatization is usually conducted with diazomethane which is a rather explosive substance. However, once the derivatives are formed, chromatographic separation is trivial and mass spectral identification is usually positive. Chemical ionization is well suited for mass spectral analysis of methyl esters. If trimethylsilyl derivatives or methyl esters are formed, the GC separation should be performed using a OV-101 column and the conditions outlined in Section 6.5.1.

6.6 LEVEL 2 ANALYSIS OF WATER SAMPLES

This Level 2 plan for analysis of water samples is taken from the "Sampling and Analysis Procedures for the Survey of Industrial Effluents for Priority Pollutants," published by the Environmental Protection Agency, Cincinnati, Ohio. Figure 6-2 is a schematic diagram of the plan. The analysis is divided into three parts, the first is a direct injection of the aqueous sample for the determination of very high concentrations of organic materials and those compounds which are not amenable to the Bellar purge and trap technique. The second step is the purge and trap technique where an aqueous sample is purged with an inert gas and the water immiscible volatile organic compounds are trapped on a Tenax solid adsorbent prior to GC/MS analysis. Finally the sample is extracted, first at an alkaline pH followed by an acidic pH extraction to separate the higher boiling and water miscible organics both basic and acidic.

6.6.1 Direct Aqueous Injection GC/MS

When impurities in the water are present at very high concentration, they can be most easily determined both qualitatively and quantitatively by direct aqueous injection of the water sample. Typically a 5 microliter sample of the water is injected onto an appropriate column such as Tenax for polar compounds and Porapak Q for nonpolar compounds using the conditions given below. The direct injection technique is also useful for the analysis of extremely volatile impurities which cannot be determined by the purge and trap technique.

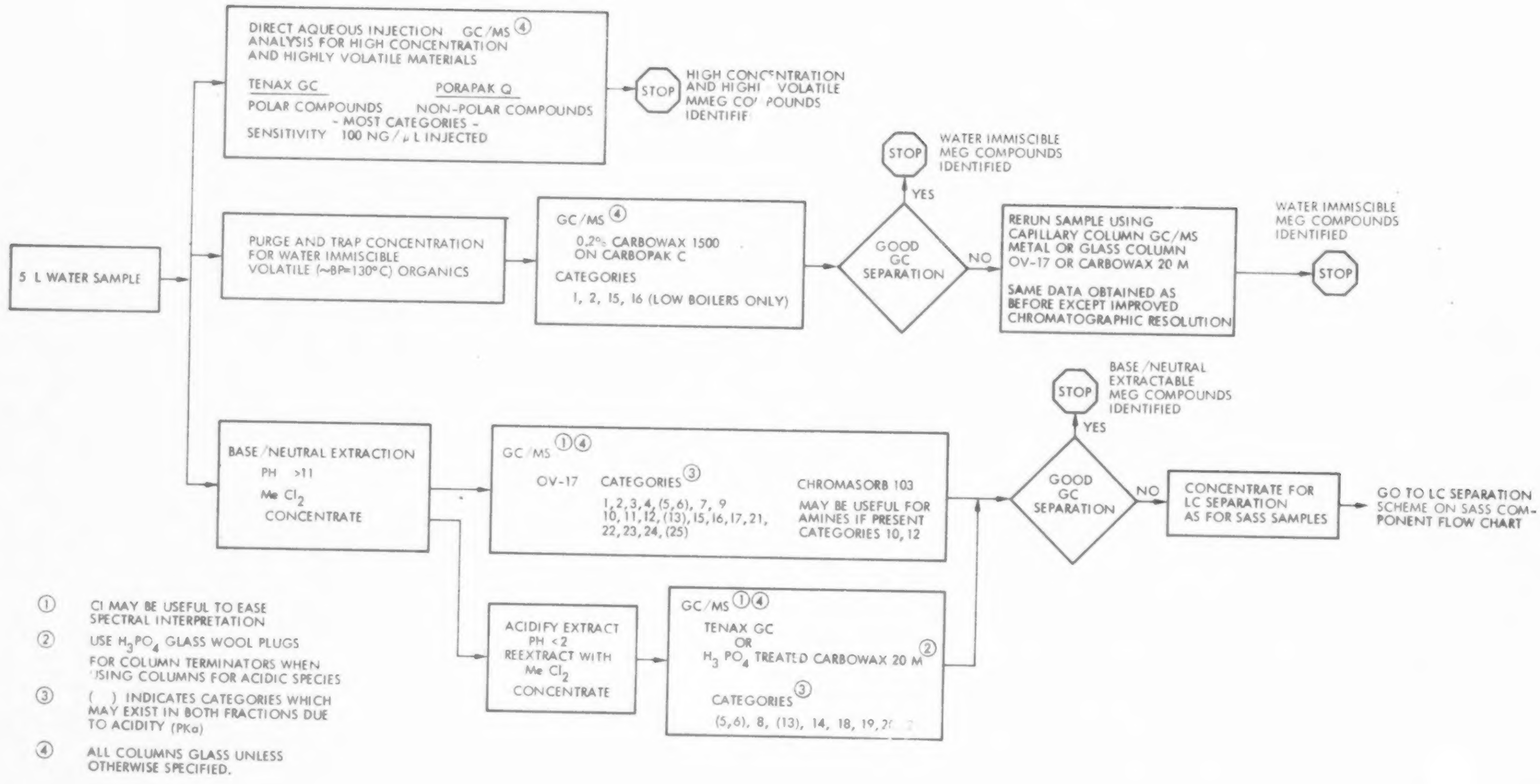

5 L WATER SAMPLE
DIRECT AQUEOUS INJECTION GC/MS ④
ANALYSIS FOR HIGH CONCENTRATION
AND HIGHLY VOLATILE MATERIALS
TENAX GC
PORAPAK Q
POLAR COMPOUNDS
NON-POLAR COMPOUNDS
- MOST CATEGORIES -
SENSITIVITY 100 NG/μL INJECTED
STOP
HIGH CONC NTRATION
AND HIGHI VOLATILE
MMEG CO POUNDS
IDENTIFI
PURGE AND TRAP CONCENTRATION
FOR WATER IMMISCIBLE
VOLATILE (~BP≈130°C) ORGANICS
GC/MS ④
0.2% CARBOWAX 1500
ON CARBOPAK C
CATEGORIES
1, 2, 15, 16 (LOW BOILERS ONLY)
GOOD
GC
SEPARATION
YES
STOP
WATER IMMISCIBLE
MEG COMPOUNDS
IDENTIFIED
NO
RERUN SAMPLE USING
CAPILLARY COLUMN GC/MS
METAL OR GLASS COLUMN
OV-17 OR CARBOWAX 20 M
SAME DATA OBTAINED AS
BEFORE EXCEPT IMPROVED
CHROMATOGRAPHIC RESOLUTION
STOP
WATER IMMISCIBLE
MEG COMPOUNDS
IDENTIFIED
BASE/NEUTRAL EXTRACTION
PH >11
Me Cl_2
CONCENTRATE
GC/MS ①④
OV-17
CATEGORIES ③
1, 2, 3, 4, (5,6), 7, 9
10, 11, 12, (13), 15, 16, 17, 21,
22, 23, 24, (25)
CHROMASORB 103
MAY BE USEFUL FOR
AMINES IF PRESENT
CATEGORIES 10, 12
ACIDIFY EXTRACT
PH <2
REEXTRACT WITH
Me Cl_2
CONCENTRATE
GC/MS ①④
TENAX GC
OR
H_3PO_4 TREATED CARBOWAX 20 M ②
CATEGORIES ③
(5,6), 8, (13), 14, 18, 19, 2
GOOD
GC
SEPARATION
YES
STOP
BASE/NEUTRAL
EXTRACTABLE
MEG COMPOUNDS
IDENTIFIED
NO
CONCENTRATE FOR
LC SEPARATION
AS FOR SASS SAMPLES
GO TO LC SEPARATION
SCHEME ON SASS COMPONENT FLOW CHART
① CI MAY BE USEFUL TO EASE SPECTRAL INTERPRETATION
② USE H_3PO_4 GLASS WOOL PLUGS FOR COLUMN TERMINATORS WHEN USING COLUMNS FOR ACIDIC SPECIES
③ ( ) INDICATES CATEGORIES WHICH MAY EXIST IN BOTH FRACTIONS DUE TO ACIDITY (PKa)
④ ALL COLUMNS GLASS UNLESS OTHERWISE SPECIFIED.

Figure 6-2. Logic Flow Chart for Level 2 Organic Aqueous Samples

Tenax GC

GC conditions for Tenax have been previously given.

Porapak Q

Porapak Q is a porous polymer which is a gas solid absorbent, and will elute most nonpolar compounds with good resolution.

- Column type: Porapak Q
- Column length: 4 mm ID by 2M long
- Temperature program: Room temperature to 240°C at 6°C per minute. Hold at maximum until all peaks elute.
- Sample size: 3 to 10 μl
- Sensitivity: 100 nanograms per μl injected.

6.6.2 Purge and Trap Concentration Technique

The purge and trap technique is designed to concentrate those organic compounds from water which are immiscible and having a boiling range up to about 130°C, very low boiling immiscible materials are not trapped by this technique. The apparatus used for this analysis consists of a purging chamber in which the sample is placed. The chamber is purged with an inert gas such as helium at a flow rate of 40 cc per minute. The purge time is approximately 12 minutes and the organic vapors are trapped on a Tenax and silica gel column which is subsequently heated and the desorbed gases injected into a gas chromatograph followed by separation on a carbowax 1500 column.

- Liquid phase: 0.2 percent Carbowax 1500
- Solid support: Carbopak C
- Column type: glass
- Column size: 2 mm ID by 3 meters long proceeded by a short column of 3 percent Carbowax 1500 on Chromasorb W
- Helium flow rate: 30 cc per minute
- Temperature program: room temperature during trap desorption followed by rapid heating to 60°C hold for 4 minutes then program at 8°C per minute to 170°C, and hold for 12 minutes or until all compounds have eluted.

- Sensitivity: variable depending on trapping efficiency, must be determined daily when analysis technique is used.

The column used in this analysis has very high resolution for nonpolar materials which are low boiling. These include categories 1, 2, 15, and 16. If the sample is highly contaminated and chromatographic resolution is insufficient for compound identification, a capillary column, either OV-17 or Carbowax 20M, may be used as a substitute in this analysis.

When using the purge and trap technique, it is necessary to run blank water samples between each analysis sample. It is also necessary to bake the trap during the course of the GC run to remove all possible interfering organic substituents which may cross over from one sample to the next due to insufficient trap heating.

6.6.3 Extraction of Water Sample for GC/MS Analysis

The extraction of water samples for subsequent analysis by GC/MS is identical to the procedure outlined for the condensate sample from the SASS train. If the chromatographic analysis of the extracts is complex and incomplete compound analysis results, the LC fractionation step should be implemented as outlined in Figure 6-1.

APPENDIX A:

LEVEL 1 DATA REDUCTION AND DECISION CHARTS

Level 1 Data Reduction and Decision Charts

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CATEGORY	COMPOUND	MATE AIR $\mu g/m^3$ (ppm)	MATE WATER $\mu g/l$ HEALTH	MATE WATER $\mu g/l$ ECOLOGY	MATE LAND $\mu g/g$ HEALTH	MATE LAND $\mu g/g$ ECOLOGY	WHERE FOUND IN LEVEL I	SAMPLE $\mu g/m^3$ $\mu g/g$ $\mu g/l$	RATIO $\frac{\text{SAMPLE}}{\text{MATE}}$	LEVEL 2 REQUIRED Y=YES N=NO	TEST METHOD[1]	TEST EXPEC-TATIONS[2]	TEST COST[3]	SAMPLE ALIQUOT[4]
1. ALIPHATIC HYDROCARBONS	Hexanes	3.6×10^5 (100)	5.4×10^6	1.0×10^5	1.1×10^4	$<2.0 \times 10^2$	GAS, FIELD, C6							
A. ALKANES AND CYCLIC ALKANES	Nonanes	1.05×10^6 (200)	1.57×10^7	N	3.2×10^4	N	SASS, LAB, C9							
	Cyclohexane	1.05×10^6 (300)	1.58×10^7	1.0×10^3	3.2×10^4	2.0	GAS, FIELD, C6							
	Butanes	1.4×10^6 (600)	2.1×10^7	$<1.0 \times 10^5$	4.2×10^4	$<2.0 \times 10^2$	GAS, FIELD, C4							
	Octanes	1.45×10^6 (300)	2.18×10^7	N	4.4×10^4	N	SASS, LAB, C8							
	Heptanes	1.6×10^6 (400)	2.4×10^7	1.0×10^5	4.8×10^4	2.0×10^2	GAS, LAB, C7							
	Pentanes	1.8×10^6 (600)	2.7×10^7	1.0×10^3	5.4×10^4	2.0	GAS, FIELD, C5							
	Methane	3.3×10^6 (5000)	4.91×10^7	$<1.0 \times 10^5$	9.8×10^4	$<2.0 \times 10^2$	GAS, FIELD, C1							
	Ethane	6.12×10^6 (5000)	9.18×10^7	N	1.8×10^5	N	GAS, FIELD, C2							
	Propane	9.0×10^6 (5000)	1.35×10^8	$<1.0 \times 10^5$	2.8×10^5	$<2.0 \times 10^2$	GAS, FIELD, C3							
	Cyclopetane	N	N	$<1.0 \times 10^5$	1.1×10^4	$<2.0 \times 10^2$	GAS, FIELD, C5							
	Alkanes (C>9)	N	N	N	N	N	SAS, LAB, C10-12, LC1							
B. ALKENES, CYCLIC Alkenes and Dienes	Cyclopentadienes	2.0×10^5 (75)	3.0×10^6	N	6.0×10^3	N	GAS, FIELD, C5							
	Cyclohexene	1.0×10^6 (300)	1.5×10^7	N	3.0×10^4	N	GAS, FIELD, C6							
	Hexenes	1.02×10^6 (300)	1.53×10^7	N	N	N	GAS, FIELD, C6							
	Butadienes	2.2×10^6 (1000)	3.3×10^7	1.0×10^3	6.6×10^4	2.0	GAS, FIELD, C3							
	Ethylene	5.71×10^6 (5000)	8.57×10^7	1.0×10^4	1.7×10^5	2.0×10	GAS, FIELD, C1							
	Propylene	8.59×10^6 (5000)	1.29×10^8	1.0×10^5	2.6×10^5	2.0×10^2	GAS, FIELD, C2							
	Butylenes	N	N	N	N	N	GAS, FIELD, C3							
	Pentenes	N	N	N	N	N	GAS, FIELD, C5							
	Cyclohexadiene	N	N	N	N	N	GAS, FIELD, C5							
	Heptenes	N	N	1.0×10^5	N	2.0×10^2	GAS, FIELD, C6							
C. ALKYNES	Propyne	1.65×10^6 (1000)	2.48×10^7	N	5.0×10^4	N	GAS, FIELD, C3							
	Acetylene	5.31×10^6 (5000)	7.97×10^7	N	1.6×10^5	N	GAS, FIELD, C2							
	Butyne	N	N	N	N	N	GAS, FIELD, C3							
2. HALOGENATED ALIPHATIC HYDROCARBON	Hexachlorocyclohexane (Lindone)	5.0×10^2	7.50×10^3	1.0×10^2	1.5×10^1	2.0×10^{-1}	SASS, LAB, C11							
A. SATURATED ALKYL HALIDES	Methyl iodide	8.54×10^2	1.28×10^4	N	2.6×10^1	N	GAS, FIELD, C5							

TABLE KEY:

1. TEST METHOD
   1. STANDARD
   2. DEVELOPMENTAL
   3. UNKNOWN

   A. AAS
   B. XRD
   C. WET CHEMICAL
   D. ESCA
   E. GC/MS
2. EXPECTED TEST SUCCESS:
   1. HIGH
   2. MODERATE
   3. UNKNOWN
3. TEST COST
   1. REASONABLE
   2. MODERATE
   3. HIGH
4. SAMPLE ALIQUOT
   1. ADEQUATE
   2. MARGINAL
   3 INADEQUATE
   4. RESAMPLE

Level 1 Data Reduction and Decision Charts (Continued)

Level 1 Data Reduction and Decision Charts (Continued)

CATEGORY	COMPOUND	MATE AIR µg/m³ (ppm)	MATE WATER µg/l HEALTH	MATE WATER µg/l ECOLOGY	MATE LAND µg/g HEALTH	MATE LAND µg/g ECOLOGY	WHERE FOUND IN LEVEL I	SAMPLE µg/m³ µg/g µg/l	RATIO SAMPLE/MATE	LEVEL 2 REQUIRED Y=YES N=NO	TEST METHOD[1]	TEST EXPEC-TATIONS[2]	TEST COST[3]	SAMPLE ALIQUOT[4]
	Tribromomethane	5.0×10^3 (0.5)	7.5×10^4	N	1.5×10^2	N	SASS, LAB, C10							
	Hexachloroethane	1.0×10^4 (1)	1.5×10^5	N	3.0×10^2	N	SASS, LAB, C11							
	1,1,2-Trichloroethane	4.5×10^4 (10)	6.75×10^5	1.0×10^3	1.4×10^3	2.0	GAS, FIELD, C5							
	Carbon tetrachloride	5.98×10^4	8.97×10^5	1.0×10^3	1.8×10^3	2.0	GAS, FIELD, C5							
	Methyl bromide	6.0×10^4 (15)	9.0×10^5	$>1.0 \times 10^5$	1.8×10^3	72.0×10^2	GAS, FIELD, C4							
	Chloroform	1.2×10^5 (25)	6.0×10^5	N	1.2×10^3	N	GAS, FIELD, C6							
	1,2-Dichloroethane	2.0×10^5 (50)	3.0×10^6	1.0×10^4	6.0×10^3	2.0×10^1	GAS, FIELD, C5							
	Methyl chloride	2.1×10^5 (100)	3.15×10^6	$>1.0 \times 10^5$	6.4×10^3	2.0×10^2	GAS, FIELD, C3							
	Dichloropropanes	3.5×10^5 (75)	5.25×10^6	1.0×10^3	1.1×10^4	2.0	GAS, FIELD, C6							
	Dichloromethane	7.2×10^5 (200)	1.08×10^7	4.5×10^3			SASS, LAB, C8							
	Bromochloromethane	1.1×10^6 (200)	1.7×10^7	N	3.4×10^4	N	GAS, FIELD, C6							
	1,2-Dichloro-1,2-difluoroethane	4.95×10^6 (1000)	7.43×10^7	N	1.5×10^5	N	GAS, FIELD, C5							
	Dichlorodifluoromethane	4.95×10^6 (1000)	7.43×10^7	$>1.0 \times 10^5$	1.5×10^5	72.0×10^2	GAS, FIELD, C3							
	Trichlorofluoromethane	5.6×10^6	8.4×10^6	N	1.7×10^5	N	GAS, FIELD, C4							
	Bromodichloromethane	N	N	N	N	N	GAS, FIELD, C6							
	Dibromodichloromethane	N	N	N	N	N	GAS, FIELD, C8							
	Bromobutanes	N	N	N	N	N	SASS, LAB, C7							
	1-Chlorooctane	N	N	N	N	N	SASS, LAB, C11							
B. UNSATURED ALKYL HALIDES	Hexachlorocyclopentadiene	1.1×10^2 (0.01)	1.65×10^3	N	3.4	N	SASS, LAB, LC2							
	Chloroethene	2.55×10^3 (1)	3.83×10^4	$>71.0 \times 10^5$	7.6×10^1	2.0×10^2	GAS, FIELD, C3							
	Hexachlorobutadiene	4.0×10^3	6.07×10^4	N	1.2×10^2	N	SASS, LAB, LC2							
	Dichloropropenes	1.12×10^4	1.68×10^5	1.0×10^3	3.4×10^2	2.0	GAS, FIELD, C6							
	1,1-Dichloroethene	2.59×10^5	3.88×10^6	4.5×10^3			GAS, FIELD, C5							
	Tetrachloroethene	6.7×10^5 (100)	1.01×10^7	1.0×10^3	2.0×10^4	2.0	SASS, LAB, C8							
	1,2-Dichloroethene	7.0×10^5 (200)	1.10×10^7	1.0×10^4	2.2×10^4	2.0×10^1	GAS, FIELD, C5							
3. ETHERS	2-Ethyl-4-Methyl-1,3-Dioxolanes	2.25×10^4	3.38×10^5	N	6.8×10^2	N	SASS, LAB, C8							
	1,3-Dioxane	1.8×10^5 (50)	2.7×10^6	N	5.4×10^3	2.0×10^1	SASS, LAB, C7							
	1,4-Dioxane	1.8×10^5	2.7×10^6	1.0×10^4	5.4×10^3	2.0×10^1	SASS, LAB, C7							
	Isopropyl ether	1.05×10^6 (250)	1.58×10^7	1.0×10^4	3.2×10^4	2.0×10^1	SASS, LAB, C7							
	2-Methoxybiphenyl	N	N	N	N	N	SASS, LAB, C7							

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   2. MODERATE
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3. TEST COST
   1. REASONABLE
   2. MODERATE
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4. SAMPLE ALIQUOT
   1. ADEQUATE
   2. MARGINAL
   3. INADEQUATE
   4. RESAMPLE

Level 1 Data Reduction and Decision Charts (Continued)

Level 1 Data Reduction and Decision Charts (Continued)

CATEGORY	COMPOUND	MATE AIR μg/m^3 (ppm)	MATE WATER μg/l HEALTH	MATE WATER μg/l ECOLOGY	MATE LAND μg/g HEALTH	MATE LAND μg/g ECOLOGY	WHERE FOUND IN LEVEL 1	SAMPLE μg/m^3 μg/g μg/l	RATIO $\frac{\text{SAMPLE}}{\text{MATE}}$	LEVEL 2 REQUIRED Y=YES N=NO	TEST METHOD[1]	TEST EXPECTATIONS[2]	TEST COST[3]	SAMPLE ALIQUOT[4]
4. HALOGENATED ETHERS	1,1-Dichlorodiethyl ether	3.0×10^4 (5)	4.5×10^5	N	9.0×10^2	N	SASS, LAB, C7							
	1,2-Dichloroethyl ether	3.0×10^4 (5)	4.5×10^5	4.5×10^3	N	N	SASS, LAB, C9							
	2,2-Dichlorodiethyl ether	3.0×10^4 (5)	4.5×10^5	1.0×10^4	9.0×10^2	2.0×10^1	SASS, LAB, C10							
	Chloromethyl methyl ether	3.68×10^4	5.52×10^5	4.5×10^3	1.1×10^3	9.0	GAS, FIELD, C5							
	1,1-Dichloromethyl ether	N	N	N	N	N	SASS, LAB, C7							
	2-Chloro-1,2 epoxypropane	N	N	N	N	N	SASS, LAB, LC4							
	2-Chloroethyl methyl ether	N	N	N	N	N	GAS, FIELD, C6							
	1-Chloro-1,2-oxetane	N	N	N	N	N	SASS, LAB, C7							
	Chloroethylethyl ether	N	N	N	N	N	SASS, LAB, C7							
	1,2-Dichloroethyl ethyl ether	N	N	N	N	N	SASS, LAB, C9							
	α-Chlorobutylethyl ether	N	N	N	N	N	SASS, LAB, C10							
	bis-(1-Chloroisopropyl) ether	N	N	N	N	N	SASS, LAB, C11							
	Bromophenyl phenyl ether	N	N	N	N	N	SASS, LAB, LC4							
5. ALCOHOLS A. PRIMARY ALCOHOLS	α-Hydroxytholyene	5.5×10^{-4}	8.3×10^5	1.0×10^4	1.7×10^3	2.0×10^1	SASS, LAB, C12							
	Isobutylalcohol	1.5×10^5 (50)	2.3×10^6	1.0×10^4	4.5×10^3	2.0×10^1	SASS, LAB, C7							
	Pentanols (Primary)	3.6×10^5 (100)	5.4×10^6	1.0×10^4	1.1×10^4	2.0×10^1	SASS, LAB, C8							
	1-Propanol	5.0×10^5	7.5×10^6	1.0×10^4	1.5×10^4	2.0×10^1	GAS, FIELD, C6							
	Butanols (Primary) N-Butanol	1.5×10^5 (50)	2.25×10^6	$>1.0 \times 10^5$	4.5×10^3	2.0×10^2	SASS, LAB, C8							
	Methanol	2.6×10^5 (200)	3.9×10^6	$>1.0 \times 10^5$	7.8×10^3	2.0×10^2	GAS, FIELD, C6							
	Ethanol	1.9×10^6 (1000)	2.85×10^7	$>1.0 \times 10^5$	5.8×10^4	2.0×10^2	GAS, FIELD, C6							
B. SECONDARY ALCOHOLS	Phenyl Ethanol	1.8×10^4	2.7×10^5	N	5.4×10^2	N	SASS, LAB, LC6							
	Benzyl	5.54×10^4	8.31×10^5	1.0×10^4			SASS, LAB, LC6							
	Borneol	9.0×10^4	1.35×10^6	N	4.8×10^3	N	SASS, LAB, LC6							
	2,6-Dimethyl-4-heptanol	1.6×10^5	2.4×10^6	N	4.8×10^3	N	SASS, LAB, C6							

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Level 1 Data Reduction and Decision Charts (Continued)

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CATEGORY	COMPOUND	MATE AIR $\mu g/m^3$ (ppm)	MATE WATER $\mu g/l$ HEALTH	MATE WATER $\mu g/l$ ECOLOGY	MATE LAND $\mu g/g$ HEALTH	MATE LAND $\mu g/g$ ECOLOGY	WHERE FOUND IN LEVEL I	SAMPLE $\mu g/m^3$ $\mu g/g$ $\mu g/l$	RATIO $\frac{\text{SAMPLE}}{\text{MATE}}$	LEVEL 2 REQUIRED Y=YES N=NO	TEST METHOD[1]	TEST EXPEC-TATIONS[2]	TEST COST[3]	SAMPLE ALIQUOT[4]
	Pentonols (secondary)	3.6×10^5 (100)	5.4×10^6	N	1.1×10^4	N	SASS, LAB, C8							
	2-Butanol	4.8×10^5 (150)	6.75×10^6	$>1.0 \times 10^5$	1.4×10^4	2.0×10^2	GAS, FIELD, C6							
	2-Propanol	9.8×10^5 (400)	1.47×10^7	1.0×10^4	3.0×10^4	2.0×10	GAS, FIELD, C6							
C. TERTIARY ALCOHOLS	Tertiary Pentanol	4.5×10^4	6.75×10^5	$>1.0 \times 10^5$	1.4×10^3	2.0×10^2	GAS, FIELD, C6							
	a-Terpineol	1.95×10^5	2.89×10^6	N	5.8×10^3	N	SASS, LAB, LC6							
	Tertiary Butanol	3.0×10^5 (100)	4.5×10^6	$>1.0 \times 10^5$	9.0×10^3	2.0×10^2								
	Isoborneol	N	N	N	N	N	SASS, LAB, LC6							
6. GLYCOLS, EPOXIDES A. GLYCOLS	Ethylene glycol	1.0×10^4	1.5×10^5	1.0×10^4	3.0×10^2	2.0×10^1	SASS, LAB, C11							
	Propylene glycol	3.6×10^5 (100)	5.4×10^6	$>1.0 \times 10^5$	1.1×10^4	2.0 x 102	SASS, LAB, C11							
B. EPOXIDES	1-Chloro-2,3-epoxypropane	1.63×10^4	2.45×10^5	1.0×10^3	4.8×10^2	2.0	SASS, LAB, C8							
	2,3-Epoxy-1-propanol	1.5×10^4 (50)	2.3×10^5	N	5.2×10^3	N	SASS, LAB, C10							
7. ALDEHYDES, KETONES A. ALDEHYDES	Acrolein	2.5×10^2 (0.1)	3.75×10^3	$<1.0 \times 10^2$	7.5	2.0×10^{-1}	GAS, FIELD, C5							
	Formaldehyde	1.6×10^3	2.4×10^4	1.0×10^3	4.8×10^1	2.0	GAS, FIELD, C3							
	Propionaldehyde	3.6×10^4	5.4×10^5	1.0×10^4	1.1×10^3	2.0×10	GAS, FIELD, C5							
	Benzaldehyde	5.83×10^4	8.77×10^5	N	1.8×10^3	N	SASS, LAB, C10							
	Butyraldehyde	1.12×10^5	1.68×10^6	1.0×10^2	3.3×10^3	2.0×10^{-1}	GAS, FIELD, C6							
	Acetaldehyde	1.8×10^5 (100)	2.7×10^6	N	5.4×10^3	N	GAS, FIELD, C4							
	3-Methylbutanal	3.86×10^5	5.79×10^6	1.0×10^3	1.2×10^4	2.0	GAS, FIELD, C6							
B. KETONES	Camphor	1.2×10^4 (2)	1.8×10^5	N	3.6×10^2	N	SASS, LAB, LC4							
	Isophorone	2.5×10^4	3.75×10^5	N	7.5×10^2	N	SASS, LAB, LC4							
	Acetophenone	4.05×10^4	6.07×10^5	N	1.2×10^3	N	SASS, LAB, LC4							
	Acetone	2.4×10^6 (1000)	3.6×10^7	$<1.0 \times 10^5$	7.2×10^4	$>2.0 \times 10^2$	GAS, FIELD, C5							
	Butanone	5.9×10^6 (200)	8.85×10^6	$<1.0 \times 10^5$	1.8×10^4	$>2.0 \times 10^2$	GAS, FIELD, C6							
	Tetrachloro-acetone	N	N	N	N	N	SASS, LAB, C11							
	Chlorohydroxy Benzophenone	N	N	N	N	N	SASS, LAB, LC4							
	5,6-Benzo-9-Anthrone	N	N	N	N	N	SASS, LAB, LC4							
	Dihydro(d)carvone	N	N	N	N	N	SASS, LAB, LC4							

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2. EXPECTED TEST SUCCESS:
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3. TEST COST
   1. REASONABLE
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4. SAMPLE ALIQUOT
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Level 1 Data Reduction and Decision Charts (Continued)

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CATEGORY	COMPOUND	MATE AIR μg/m^3 (ppm)	MATE WATER μg/l HEALTH	MATE WATER μg/l ECOLOGY	MATE LAND μg/g HEALTH	MATE LAND μg/g ECOLOGY	WHERE FOUND IN LEVEL I	SAMPLE μg/m^3 μg/g μg/l	RATIO SAMPLE MATE	LEVEL 2 REQUIRED Y=YES N=NO	TEST METHOD^1	TEST EXPEC-TATIONS^2	TEST COST^3	SAMPLE ALIQUOT^4
8. CARBOXYLIC ACIDS A. CARBOXYLIC ACIDS	Maleic	1.0×10^3 (0.25)	1.5×10^4	N	3.0×10^1	N	SASS, LAB, C8							
	Phthalic	6.0×10^3 (1.0)	9.0×10^4	N	1.8×10^2	N	SASS, LAB, LC7 & 8							
	Formic	9.0×10^3 (5.0)	1.4×10^5	N	2.7×10^2	N	SASS, LAB, C8							
	Acetic	2.5×10^4 (10)	3.8×10^5	1.0×10^3	7.6×10^2	2.0	SASS, LAB, LC7 & 8							
	Benzoic	1.4×10^5	2.1×10^6	N	4.2×10^3	N	SASS, LAB, C8, 8 & LC7							
	Long Chain	N	N	N	N	N	SASS, LAB, C8, 8 & LC7							
B. CARBOXYLIC ACIDS WITH ADDITIONAL FUNCTIONAL GROUPS	3-Hydroxypropanoic Acid Lactone	3.17×10^2	4.76×10^3	1.0×10^4	9.6	2.0×10	SASS, LAB, C10							
	β-Propiolactone	3.2×10^2	1.6×10^3	N	3.2	N								
	Hydroxybenzoic Acid	4.01×10^4	6.01×10^5	N	1.2×10^3	N	SASS, LAB, C7							
	Hydroxyacetic Acid	8.78×10^4	1.13×10^6	N	2.6×10^3	N	SASS, LAB, LC7							
C. AMIDES	6-Hevanelactam	1.0×10^3	1.5×10^4	N	3.0	N	SASS, LAB, C7							
	Formamide	3.0×10^4 (20)	4.5×10^5	N	9.0×10^2	N	SASS, LAB, LC7							
	Acetamide	4.5×10^5	6.75×10^6	N	1.4×10^4	N	SASS, LAB, LC7							
	6-Aminohexanoic Acid	2.33×10^6	3.5×10^7	N	N	N	SASS, LAB, LC8							
D. ESTERS	Phthalates	5.0×10^3	7.5×10^4	1.5	1.5×10^2	3.0×10^{-3}	SASS, LAB, LC6							
	Methyl Methacrylate	4.1×10^5 (100)	6.2×10^6	1.0×10^4	1.2×10^4	2.0×10	SASS, LAB, C7							
	Adipates	1.89×10^4	2.83×10^5	N	5.6×10^2		SASS, LAB, C8, 9 & 10							
	Methyl Benzoate	1.5×10^5	2.25×10^6	N	4.6×10^3	N	SASS, LAB, C10							
	Phenyl Benzoate	N	N	N	N	N	SASS, LAB, LC6							
	Di-2-ethylhexyl phthalate	N	N	N	N	N	SASS, LAB, LC6							
	Long chain esters	N	N	N	N	N	SASS, LAB, LC6							
9. NITRILES	1-Cyanoethane	1.76×10^3	2.64×10^4	N	5.4×10	N	GAS, FIELD, C6							
	Tetramethyl-succinonitrile	3.0×10^3 (0.5)	4.5×10^4	N	9.0×10	N	SASS, LAB, LC4							
	Butyronitrile	2.25×10^4	3.37×10^5	N	6.8×10^2	N	SASS, LAB, LC4							
	Benzonitrile	3.24×10^4	4.86×10^5	N	1.4×10^3	2.0	SASS, LAB							
	Acrylonitrile	4.5×10^4 (20)	6.8×10^5	1.0×10^3	1.4×10^3		SASS, LAB, C11							
	Acetonitrile	7.0×10^4 (40)	1.05×10^6	1.0×10^5	2.1×10^3	2.0×10^2	GAS, FIELD, C6							
	Naphthonitriles	N	N	N	N	N	SASS, LAB, LC4							

TABLE KEY:

1. TEST METHOD
   1. STANDARD
   2. DEVELOPMENTAL
   3. UNKNOWN
   - A. AAS
   - B. XRD
   - C. WET CHEMICAL
   - D. ESCA
   - E. GC/MS
2. EXPECTED TEST SUCCESS:
   1. HIGH
   2. MODERATE
   3. UNKNOWN
3. TEST COST
   1. REASONABLE
   2. MODERATE
   3. HIGH
4. SAMPLE ALIQUOT
   1. ADEQUATE
   2. MARGINAL
   3. INADEQUATE
   4. RESAMPLE

Level 1 Data Reduction and Decision Charts (Continued)

Level 1 Data Reduction and Decision Charts (Continued)

CATEGORY	COMPOUND	MATE AIR $\mu g/m^3$ (ppm)	MATE WATER $\mu g/l$ HEALTH	MATE WATER $\mu g/l$ ECOLOGY	MATE LAND $\mu g/g$ HEALTH	MATE LAND $\mu g/g$ ECOLOGY	WHERE FOUND IN LEVEL I	SAMPLE $\mu g/m^3$ $\mu g/g$ $\mu g/l$	RATIO $\frac{\text{SAMPLE}}{\text{MATE}}$	LEVEL 2 REQUIRED Y=YES N=NO	TEST METHOD[1]	TEST EXPECTATIONS[2]	TEST COST[3]	SAMPLE ALIQUOT[4]
10. AMINES	1-Aminonaphtalene	5.5×10^2	8.5×10^3	1.0×10^2			SASS, LAB, LC7							
A. PRIMARY	Methylamine	1.81×10^3	2.72×10^4	N	3.6×10	2.0	SASS, LAB, LC6							
	4-Aminibiphenyl	1.2×10^3 (10)	1.8×10^4	1.0×10^3			GAS, FIELD, C6							
	Ethanolamine	6.0×10^3 (3)	9.0×10^4	1.0×10^4	1.8×10^2	2.0×10	SASS, LAB, C10							
	Butylamines	1.5×10^4 (5)	2.25×10^5	1.0×10^5	4.5×10^2	2.0×10^2	GAS, FIELD, C6							
	Ethylamine	1.8×10^4 (10)	2.7×10^5	1.0×10^3	5.4×10^2	2.0	GAS, FIELD, C4							
	1,2-Diaminoethane	2.5×10^4 (10)	3.75×10^5	1.0×10^3	7.6×10^2	2.0	SASS, LAB, C8							
	Cyclohexylamine	4.0×10^4 (10)	5.0×10^5	1.0×10^4	1.2×10^3	2.0×10	SASS, LAB, C8							
	Propanolamine	1.27×10^5	1.9×10^6	N	3.8×10^3	N	SASS, LAB, C9							
	3-Aminopropene	N	N	N	N	N	GAS, FIELD, C5							
B. SECONDARY	Ethyleneimine	3.33×10^2	5.0×10^3	N	1.0×10	N	GAS, FIELD, C3?							
	Dimethylamine	1.8×10^4 (10)	2.7×10^5	1.0×10^3	5.4×10^2	2.0	GAS, FIELD, C3?							
	Morpholine	7.0×10^4 (20)	1.05×10^6	1.0×10^4	2.1×10^3	2.0×10	SASS, LAB, LC6?							
	Diethylamine	7.5×10^4 (25)	1.13×10^6	1.0×10^3	2.2×10^3	2.0	GAS, FIELD, C3?							
	Ethylmethylamine	N	N	N	N	N	GAS, FIELD, C4?							
C. TERTIARY AMINES	Aminotoluenes	1.1×10^2	1.7×10^3	N	3.0	N	SASS, LAB, C11							
	2-Aminonaphthalene	1.7×10^2	2.5×10^3	1.0×10^2	5.0	2.0×10^{-1}	SASS, LAB, LC6							
	4,4^{1}-Methylene-bis-(2-chloro-aniline	2.18×10^2	3.27×10^3	N	6.0	N	SASS, LAB, LC7							
	Anisidines	5.0×10^2 (0.1)	7.5×10^3	N	1.5×10	N	SASS, LAB, LC6							
	4-Aminibiphenyl	1.3×10^3	2.0×10^4	N	4.0	N	SASS, LAB, LC6							
	1,4-Diaminobenzene	4.5×10^3	6.75×10^4	N	1.4×10^3	N	SASS, LAB, LC6							
	3,3'-Dichloro-benzidine	6.6×10^3	9.9×10^4	N	2.0×10^2	N	SASS, LAB, LC7							
	Benzidine	1.4×10^4	2.1×10^5	1.0×10^2	4.2×10^2	2.0×10^{-1}	SASS, LAB, LC7							
	Aniline	1.9×10^4 (5)	2.85×10^5	1.0×10^3	6.0×10^2	2.0	SASS, LAB, C11							
	Dimethylaniline	2.5×10^4 (5)	3.75×10^5	N	7.5×10^2	N	SASS, LAB, C11							
	N,N-Dimethyl-aniline	2.5×10^4 (5)	3.83×10^5	N	7.5×10^2	N	SASS, LAB, LC6							
11. AZO COMPOUNDS	N,N'-Dimethyl-hydrazine	3.17×10	4.76×10^2	N	1.0	N	GAS, FIELD, C6							
	Monoethyl-hydrazine	3.5×10^2 (0.2)	5.25×10^3	N	1.1×10	N	GAS, FIELD, C6							
	Diazomethane	4.0×10^2 (0.2)	6.0×10^3	N	1.2×10	N	GAS, FIELD, C3							
	N,N-Dimethyl-hydrazine	1.0×10^3 (0.5)	1.5×10^4	N	3.0×10	N	SASS, LA3, C8							
	Hydrazobenzene	1.35×10^4	2.02×10^5	N			GAS, FIELD, C6							
	Diphenylhydrazines	1.4×10^4	2.0×10^5	N	4.0×10^2	N	SASS, LAB, LC7							
	Hydrazine	N	N	N			SASS, LAB, C8							

TABLE KEY:

1. TEST METHOD
   1. STANDARD
   2. DEVELOPMENTAL
   3. UNKNOWN

   A. AAS
   B. XRD
   C. WET CHEMICAL
   D. ESCA
   E. GC/MS
2. EXPECTED TEST SUCCESS
   1. HIGH
   2. MODERATE
   3. UNKNOWN
3. TEST COST
   1. REASONABLE
   2. MODERATE
   3. HIGH
4. SAMPLE ALIQUOT
   1. ADEQUATE
   2. MARGINAL
   3. INADEQUATE
   4. RESAMPLE

Level 1 Data Reduction and Decision Charts (Continued)

Level 1 Data Reduction and Decision Charts (Continued)

CATEGORY	COMPOUND	MATE AIR -g/m^3 (ppm)	MATE WATER -g/l HEALTH	MATE WATER -g/l ECOLOGY	MATE LAND -g/g HEALTH	MATE LAND -g/g ECOLOGY	WHERE FOUND IN LEVEL I	SAMPLE -g/m^3 µg/g µg/l	RATIO $\frac{\text{SAMPLE}}{\text{MATE}}$	LEVEL 2 REQUIRED Y=YES N=NO	TEST METHOD[1]	TEST EXPECTATIONS[2]	TEST COST[3]	SAMPLE ALIQUOT[4]
12. NITROSAMIES	N-Nitroso-Dimethylamine	1.2	1.8 x 10	N	3.6 x 10^{-2}	N	SASS, LAB, C9							
	N-Nitroso-Diethylamine	1.21 x 10^2	1.82 x 10^3	N	3.6	N	SASS, LAB, C10							
	N-Methyl-N-Nitroso-Amiline	1.29 x 10^3	1.94 x 10^4	N	3.8 x 10	N	SASS, LAB, LC5							
	N-Nitroso-Di-propylaime	2.41 x 10^3	3.62 x 10^4	N	7.2 x 10	N	SASS, LAB, C12 & LC5							
	N-Nitroso-Di-phenylamine	7.4 x 10^4	1.1 x 10^6	N	2.2 x 10^3	N	SASS, LAB, LC5							
	N-Nitroso-Diiso-propylamine	3.83 x 10^4	5.75 x 10^5	N	1.2 x 10^3	N	SASS, LAB, C11							
	N-Nitroso-Di-pentylamine	N	N	N	N	N	SASS, LAB, LC5							
13. MERCAPTANS, SULFIDES A. MERCAPTANS	Perchloromethyl Mercaptan	8.0 x 10^2 (0.1)	1.2 x 10^4	N	2.4 x 10	N	SASS, LAB, LC6							
	Methyl Mercaptan	1.0 x 10^3 (0.5)	1.5 x 10^4	N	3.0 x 10	N	GAS, FIELD, SGC							
	Ethyl Mercaptan	1.0 x 10^3 (0.5)	1.5 x 10^4	N	3.0 x 10	N	GAS, FIELD, SGC							
	Butyl Mercaptans	1.5 x 10^3 (0.5)	2.25 x 10^4	N	4.5 x 10	N	GAS, FIELD, SGC							
	Benzenthiol	2.07 x 10^3	3.10 x 10^4	N	5.2 x 10	N	SASS, LAB, LC6							
	Propyl Mercaptans	8.06 x 10^4	1.21 x 10^6	N	2.4 x 10^3	N	GAS, FIELD, SGC							
	1-Anthranthiol	N	N	N	N	N	SASS, LAB, LC6							
B. SULFIDES	Dimethyl Sulfide	2.41 x 10^4	3.61 x 10^5	N	7.2 x 10^2	N	GAS, FIELD, SGC							
DISULFIDES	Phenyl Sulfide	9.63 x 10^4	1.44 x 10^6	N	2.9	N	SASS, LAB, C9 & LC5							
	Diethyl Sulfide	N	N	N	N	N	GAS, FIELD, SGC							
	Methyl Disulfide	N	N	N	N	N	SASS, LAB, C8							
14. SULFONIC ACIDS, , SULFOXIDES A. SULFONIC ACIDS	Benzensulfonic Acid	4.01 x 10^4	6.01 x 10^5	N	1.2 x 10^3	N	SASS, LAB, LC8							
	9,10-Anthraquinone-Disulfonic Acid	N	N	N	N	N	SASS, LAB, LC8							
B. SULFOXIDES	Dimethyl Sulfoxide	8.14 x 10^2	1.22 x 10^3	N	2.4	N	SASS, LAB, C11							

TABLE KEY:

1. TEST METHOD
   1. STANDARD
   2. DEVELOPMENTAL
   3. UNKNOWN

   A. AAS
   B. XRD
   C. WET CHEMICAL
   D. ESCA
   E. GC/MS
2. EXPECTED TEST SUCCESS:
   1. HIGH
   2. MODERATE
   3. UNKNOWN
3. TEST COST
   1. REASONABLE
   2. MODERATE
   3. HIGH
4. SAMPLE ALIQUOT
   1. ADEQUATE
   2. MARGINAL
   3. INADEQUATE
   4. RESAMPLE

Level 1 Data Reduction and Decision Charts (Continued)

Level 1 Data Reduction and Decision Charts (Continued)

CATEGORY	COMPOUND	MATE AIR $\mu g/m^3$ (ppm)	MATE WATER $\mu g/l$ HEALTH	MATE WATER $\mu g/l$ ECOLOGY	MATE LAND $\mu g/g$ HEALTH	MATE LAND $\mu g/g$ ECOLOGY	WHERE FOUND IN LEVEL I	SAMPLE $\mu g/m^3$ $\mu g/g$ $\mu g/l$	RATIO $\frac{\text{SAMPLE}}{\text{MATE}}$	LEVEL 2 REQUIRED Y=YES N=NO	TEST METHOD[1]	TEST EXPEC-TATIONS[2]	TEST COST[3]	SAMPLE ALIQUOT[4]
15. BENZENE, SUBSTITUTED BENZENE	Biphenyl	1.0×10^3 (0.2)	1.5×10^4	N	3.0×10	N	SASS, LAB, LC2							
	Benzene	3.0×10^3 (10)	4.5×10^4	1.0×10^3	9.0×10	2.0	GAS, FIELD, C6							
	Terphenyls	9.0×10^3 (1)	1.35×10^5	N	2.8×10^2	N	SASS, LAB, LC3							
	Indene	4.5×10^4 (10)	6.8×10^5	1.0×10^3	1.4	N	SASS, LAB, LC3							
	Isopropyl Benzene	6.3×10^4	9.45×10^5	4.5×10^2	1.9×10^3	2.0	SASS, LAB, C9							
	Trimethyl Benzenes	1.2×10^5 (25)	1.8×10^6	N	3.6×10^3	N	SASS, LAB, C10							
	Dihydronaphthalenes	1.27×10^5	1.91×10^6	1.0×10^3	4.0×10^3	2.0	SASS, LAB, LC2							
	Tetrahydronaphthalenes	1.29×10^5	1.94×10^6	1.0×10^3	4.0×10^3	2.0	SASS, LAB, C12, LC2							
	Propyl Benzene	2.17×10^5	3.25×10^6	1.0×10^3	6.6×10^3	2.0	SASS, LAB, C9							
	Dialkyl Benzene	2.25×10^5	3.38×10^6	1.0×10^3	6.8×10^3	2.0	SASS, LAB, C10, 11, 12							
	Butyl Benzene	2.25×10^5	3.38×10^6	N	6.8×10^3	N	SASS, LAB, C11							
	Indane	2.3×10^5	3.4×10^6	N	6.8×10^3	N	SASS, LAB, LC2							
	Toluene	3.75×10^5 (100)	5.63×10^6	1.0×10^3	1.1×10^4	2.0	SASS, LAB, C8							
	Styrene	4.2×10^5 (100)	6.3×10^6	1.0×10^3	1.3×10^4	2.0	SASS, LAB, C9							
	Ethyl Benzene	4.35×10^5 (100)	6.53×10^6	1.0×10^3	1.3×10^4	2.0	SASS, LAB, C8							
	Xylenes	4.35×10^5 (100)	6.53×10^6	1.0×10^3	1.3×10^4	2.0	SASS, LAB, C9							
	4,4'-Diphenylbiphenyl	N	N	N	N	N	SASS, LAB, LC2 & 3							
	Tetramethyl Benzenes	N	N	1.0×10^4	N	2.0×10	SASS, LAB, C11							
16. HALOGENATED AROMATIC HYDROCARBONS A. RING SUBSTITUED AROMATICS	Polychlorinated biphenyls	5.0×10^2	7.5×10^3	5.0×10^{-3}			SASS, LAB, LC2 & 3							
	Polychlorinated benzenes	3.4×10^4	5.1×10^5	1.0×10^2	1.0×10^3	2.0×10^{-1}	SASS, LAB, LC2 & 3							
	Chloronaphthalenes	6.93×10^4	1.04×10^6	N	1.5×10	1.0×10^{-5}	SASS, LAB, LC2 & 3							
	2-Chlorotoluene	2.5×10^5 (50)	3.75×10^6	N	7.5×10^3	N	SASS. :AB. C9							
	1,2-Dichlorobenzene	3.0×10^5 (50)	4.5×10^6	1.0×10^2	9.0×10^3	2.0×10^{-1}	SASS, LAB, C10							
	Chlorobenzene	3.5×10^5 (75)	5.25×10^6	1.0×10^2	1.1×10^4	2.0×10^{-1}	SASS, LAB, C8							
	1,4-Dichlorobenzene	4.75×10^5 (76)	6.8×10^6	1.0×10^2	1.4×10^4	2.0×10^{-1}	SASS, LAB, C10							

TABLE KEY:

1. TEST METHOD:
   1. STANDARD
   2. DEVELOPMENTAL
   3. UNKNOWN

   A. AAS
   B. XRD
   C. WET CHEMICAL
   D. ESCA
   E. GC/MS

2. EXPECTED TEST SUCCESS:
   1. HIGH
   2. MODERATE
   3. UNKNOWN

3. TEST COST
   1. REASONABLE
   2. MODERATE
   3. HIGH

4. SAMPLE ALIQUOT
   1. ADEQUATE
   2. MARGINAL
   3. INADEQUATE
   4. RESAMPLE

Level 1 Data Reduction and Decision Charts (Continued)

Level 1 Data Reduction and Decision Charts (Continued)

CATEGORY	COMPOUND	MATE AIR $\mu g/m^3$ (ppm)	MATE WATER $\mu g/l$ HEALTH	MATE WATER $\mu g/l$ ECOLOGY	MATE LAND $\mu g/g$ HEALTH	MATE LAND $\mu g/g$ ECOLOGY	WHERE FOUND IN LEVEL I	SAMPLE $\mu g/m^3$ $\mu g/g$ $\mu g/l$	RATIO $\frac{\text{SAMPLE}}{\text{MATE}}$	LEVEL 2 REQUIRED Y=YES N=NO	TEST METHOD[1]	TEST EXPEC-TATIONS[2]	TEST COST[3]	SAMPLE ALIQUOT[4]
B. AROMATICS WITH HALO-GENERATED ALKYL SIDE CHAINS	Bromo and Dibromobenzenes	N	N	N	N	N	SASS, LAB, C9, 12, & LC2							
	Bromochlorobenzenes	N	N	N	N	N	SASS, LAB, C12 & LC2							
	1,3-Dichlorobenzene	N	N	N	N	N	SASS, LAB, C10							
	α-Chlorotoluene	5.54×10^4	9.31×10^5	1.0×10^2			SASS, LAB, C11							
	Bis-(chloromethyl) Benzene	N	N	N	N	N	SASS, LAB, LC2							
17. AROMATIC NITRO COMPOUNDS	4-Nitrobiphenyl	1.3×10^3	2.0×10^4	N	4.0×10	N	SASS, LAB, LC4							
	Dinitrotoluenes	1.50×10^3	2.25×10^4	1.0×10^3	4.5×10	2.0	SASS, LAB, LC5							
	Methoxynitrobenzene	4.5×10^3	6.75×10^4	N	1.4×10^2	N	SASS, LAB, LC5							
	Nitrobenzene	5.0×10^3 (1.0)	7.5×10^4	1.0×10^3	1.5×10^2	2.0	SASS, LAB, C12 & LC4							
	1-Chloro-2-Nitrobenzene	1.3×10^4	1.95×10^5	1.0×10^4	4.0×10^2	2.0×10	SASS, LAB, LC4							
	1-Chloro-4-Nitrobenzene	1.89×10^4	2.84 x 105	N	5.8×10^2	N	SASS, LAB, LC4							
	Nitrotoluenes	3.0×10^4 (5)	4.5 x 105	1.0×10^3	9.0×10^2	2.0	SASS, LAB, LC5							
18. PHENOLS A. MONOHYDRICS	2,2'-Dihydroxydiphenyls	6.75×10^3	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, LC6							
	Polyalkyl Phenols	1.49×10^4	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, LC6							
	Phenol	1.9×10^4 (5)	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, C11							
	Cresols	2.2×10^4 (5)	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, C12 & LC6							
	Phenyl Phenols	2.3×10^4	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, LC6							
	Alkyl Cresols	2.39×10^4	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, C12 & LC6							
	2-Methoxy Phenol	3.26×10^4	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, LC6							
	Xylenols	1.3×10^4	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, LC6							
	Ethylphenols	N	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, LC6							

TABLE KEY:

1. TEST METHOD
   1. STANDARD
   2. DEVELOPMENTAL
   3. UNKNOWN

   A. AAS
   B. XRD
   C. WET CHEMICAL
   D. ESCA
   E. GC/MS

2. EXPECTED TEST SUCCESS:
   1. HIGH
   2. MODERATE
   3. UNKNOWN

4. TEST COST
   1. REASONABLE
   2. MODERATE
   3. HIGH

4. SAMPLE ALIQUOT
   1. ADEQUATE
   2. MARGINAL
   3. INADEQUATE
   4. RESAMPLE

Level 1 Data Reduction and Decision Charts (Continued)

Level 1 Data Reduction and Decision Charts (Continued)

CATEGORY	COMPOUND	MATE AIR µg/m^3 (ppm)	MATE WATER µg/l HEALTH	MATE WATER µg/l ECOLOGY	MATE LAND µg/g HEALTH	MATE LAND µg/g ECOLOGY	WHERE FOUND IN LEVEL I	SAMPLE µg/m^3 µg/g µg/l	RATIO SAMPLE/MATE	LEVEL 2 REQUIRED Y=YES N=NO	TEST METHOD[1]	TEST EXPECTATIONS[2]	TEST COST[3]	SAMPLE ALIQUOT[4]
B. DIHYDRICS, POLYHYDRICS	1,4-Dihydroxybenzene	2.0×10^3	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, LC6							
	Catehol	2.0×10^4 (5)	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, LC6							
	1,2,3-Trihydroxybenzenes	3.55×10^4	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, LC6							
	1,3-Dihydroxybenzene	4.5×10^4 (10)	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, C10							
C. FUSED RING HYDROXY COMPOUNDS	1-Naphthol	1.17×10^5	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, LC6							
	2-Naphthol	1.09×10^5	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, LC6							
	Indanols	1.46×10^5	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, LC6							
	Phenanthrols	N	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, LC6							
	Acenaphthenols	N	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, LC6							
	2-Hydroxyfluorene	N	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, LC6							
	2-Hydroxydibenzofuran	N	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, LC6							
19. HALOPHENOLS	Pentachlorophenol	5.0×10^2	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, LC7							
	Chlorinated Cresols	2.25×10^4	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, LC6							
	2,4-Dichlorophenol	7.0×10^3	5.0	5.0×10^2			SASS, LAB, C12							
	2-Chlorophenol	3.02×10^4	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, C10							
20. NITROPHENOLS	Trinitrophenol	1.0×10^2 (0.011)	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, LC7							
	Dinitro-o-cresol	2.0×10^2 (0.025)	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, LC7							
	Dinitro-p-cresol	6.8×10^2	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, LC7							
	Dinitrophenols	1.35×10^3	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, LC7							
	4-Nitrophenol	1.58×10^4	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, LC7							
	3-Nitrophenol	2.01×10^4	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, C11 & LC7							
	2-Amino-4,6-Nitrophenol	4.64×10^4	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, LC7							
	2-Nitrophenol	5.8×10^4	5.0	5.0×10^2	1.0×10^{-2}	1.0	SASS, LAB, C12 & LC7							
21. FUSED AROMATIC HYDROCARBONS	Benzo(a)pyrene	2.11×10^{-2}	3.17×10^{-1}	N	6.0×10^{-2}	N	SASS, LAB, LC3							
	Dibenzo(a,h) anthracene	9.27×10^{-2}	1.39	N	3.0×10^{-3}	N	SASS, LAB, LC3							
	7,12-Dimethylbenz-(a)-anthracene	2.6×10^{-1}	3.91	N	8.0×10^{-3}	N	SASS, LAB, LC3							
	Dibenzo(a,i)pyrene	4.3×10	6.5×10^2	N	.3	N	SASS, LAB, LC3							

TABLE KEY:

1. TEST METHOD:
   1. STANDARD
   2. DEVELOPMENTAL
   3. UNKNOWN

   A. AAS
   B. XRD
   C. WET CHEMICAL
   D. ESCA
   E. GC/MS
2. EXPECTED TEST SUCCESS:
   1. HIGH
   2. MODERATE
   3. UNKNOWN
3. TEST COST
   1. REASONABLE
   2. MODERATE
   3. HIGH
4. SAMPLE ALIQUOT
   1. ADEQUATE
   2. MARGINAL
   3. INADEQUATE
   4. RESAMPLE

Level 1 Data Reduction and Decision Charts (Continued)

Level 1 Data Reduction and Decision Charts (Continued)

CATEGORY	COMPOUND	MATE AIR $\mu g/m^3$ (ppm)	MATE WATER $\mu g/l$ HEALTH	MATE WATER $\mu g/l$ ECOLOGY	MATE LAND $\mu g/g$ HEALTH	MATE LAND $\mu g/g$ ECOLOGY	WHERE FOUND IN LEVEL I	SAMPLE $\mu g/m^3$ $\mu g/g$ $\mu g/l$	RATIO $\frac{SAMPLE}{MATE}$	LEVEL 2 REQUIRED Y=YES N=NO	TEST METHOD[1]	TEST EXPEC-TATIONS[2]	TEST COST[3]	SAMPLE ALIQUOT[4]
	9,10-Dimethyl-1,1 2-benzanthracene	2.96×10	4.44×10^2	N			SASS, LAB, LC3							
	Benz(a)anthracene	4.45×10	6.72×10^2	N	1.3	N	SASS, LAB, LC3							
	Dibenzo(b,def) Chrysene	3.32×10^2	4.98×10^3	N	N	N	SASS, LAB, LC3							
	Benzo(g,h,i) perylene	5.43×10^2	8.15×10^3	N	N	N	SASS, LAB, LC3							
	Dibenzo(a,i)pyrene	1.08×10^3	1.62×10^3	N	3.2×10	N	SASS, LAB, LC3							
	Phenanthrene	1.59×10^3	2.39×10^4	N	4.8×10	N	SASS, LAB, LC3							
	Methylchrysenes	1.79×10^3	2.69×10^4	N	5.4×10	N	SASS, LAB, LC3							
	Chrysene	2.2×10^3	3.3×10^4	N	6.6×10	N	SASS, LAB, LC3							
	Picene	2.5×10^3	3.75×10^4	N	7.5×10	N	SASS, LAB, LC3							
	Benzo(e)pyrene	3.04×10^3	4.56×10^4	N	9.1×10	N	SASS, LAB, LC3							
	Dibenzo(a,h)pyrene	3.68×10^3	5.52×10^4	N	1.1×10^2	N	SASS, LAB, LC3							
	Dibenz(a,c) anthracene	9.9×10^3	1.5×10^5	N	3.0×10^2	N	SASS, LAB, LC3							
	1,2:3,4-Dibenzo-anthracene	1.0×10^4	1.5×10^5	N			SASS, LAB, LC3							
	Benzo(g)chrysene	1.63×10^4	2.45×10^5	N	4.8×10^2	N	SASS, LAB, LC3							
	Benzo(c)phen-anthrene	2.69×10^4	4.04×10^5	N	8.2×10^2	N	SASS, LAB, LC3							
	Methylphen-anthrenes	3.04×10^4	4.56×10^5	N	9.1×10^2	N	SASS, LAB, LC3							
	Naphthalene	5.0×10^4	7.5×10^5	1.0×10^2	1.5×10^3	2.0×10^{-1}	SASS, LAB, C11 & LC2							
	Anthracene;Methyl Anthracene	5.6×10^4	8.4×10^5	N	1.7×10^3	N	SASS, LAB, LC3							
	Monoalkyl Naphthalenes	2.25×10^5	3.38×10^6	N	6.8×10^3	N	SASS, LAB, LC2							
	Dimethyl Naphthalenes	2.25×10^5	3.38×10^6	N	6.8×10^3	N	SASS, LAB, LC2							
	Pyrene	2.33×10^5	3.5×10^6	N	6.9×10^3	N	SASS, LAB, LC3							
	Phenyl Naphtha-lenes	N	N	N	N	N	SASS, LAB, LC3							
	Acenaphthene; Acenaphthylene	N	N	N	N	N	SASS, LAB, LC3							
	2,7-Dimethyl-anthracene	N	N	N	N	N	SASS, LAB, LC3							
	Naphthacene	N	N	N	N	N	SASS, LAB, LC3							
	Triphenylene	N	N	N	N	N	SASS, LAB, LC3							

TABLE KEY:

1. TEST METHOD
   1. STANDARD
   2. DEVELOP-MENTAL
   3. UNKNOWN

   A. AAS
   B. XRD
   C. WET CHEMICAL
   D. ESCA
   E. GC/MS
2. EXPECTED TEST SUCCESS:
   1. HIGH
   2. MODERATE
   3. UNKNOWN
3. TEST COST
   1. REASON-ABLE
   2. MODERATE
   3. HIGH
4. SAMPLE ALIQUOT
   1. ADEQUATE
   2. MARGINAL
   3. INADEQUATI
   4. RESAMPLE

Level 1 Data Reduction and Decision Charts (Continued)

Level 1 Data Reduction and Decision Charts (Continued)

CATEGORY	COMPOUND	MATE AIR µg/m³ (ppm)	MATE WATER µg/ℓ HEALTH	MATE WATER µg/ℓ ECOLOGY	MATE LAND µg/g HEALTH	MATE LAND µg/g ECOLOGY	WHERE FOUND IN LEVEL I	SAMPLE µg/m³ µg/g µg/l	RATIO SAMPLE/MATE	LEVEL 2 REQUIRED Y=YES N=NO	TEST METHOD[1]	TEST EXPEC-TATIONS[2]	TEST COST[3]	SAMPLE ALIQUOT[4]
	1-Methyl Pyrene	N	N	N	N	N	SASS, LAB, LC3							
	Dimethyl Pyrenes	N	N	N	N	N	SASS, LAB, LC3							
	1,2 Benzoaphthacene	N	N	N	N	N	SASS, LAB, LC3							
	Perylene	N	N	N	N	N	SASS, LAB, LC3							
	Coronene	N	N	N	N	N	SASS, LAB, LC3							
22. FUSED NON-ALTERNATE POLYCYCLIC HYDROCARBONS	3-Methyl-Cholanthrene	3.75	5.63×10	N	1.1×10^{-1}	N	SASS, LAB, LC3							
	Benzo(b) fluoranthene	8.97×10^2	1.35×10^4	N	2.8×10	N	SASS, LAB, LC3							
	Benzo(k) fluoranthene	1.63×10^3	2.45×10^4	N	4.9×10	N	SASS, LAB, LC3							
	Indeno(1,2,3,c,d) pyrene	1.63×10^3	2.45×10^4	N	4.8×10	N	SASS, LAB, LC3							
	Benzo(j) fluoranthene	6.48×10^3	9.72×10^4	N	2.0×10^2	N	SASS, LAB, LC3							
	1,2:5,6-Dibenzo-fluorene	1.32×10^4	1.98×10^5	N	4.0×10^2	N	SASS, LAB, LC3							
	Dicyclopentadiene	1.59×10^4	2.39×10^5	1.0×10^2	4.8×10^2	2.0×10^{-1}	SASS, LAB, C10							
	Indane, Indene	4.5×10^4 (10)	6.75×10^5	N	N	N	SASS, LAB, C11							
	Fluoranthene, (Tetrahydro-fluoroanthene)	9.0×10^4	1.35×10^5	N	2.8×10^3	N	SASS, LAB, LC3							
	Fluorene	N	N	N	N	N	SASS, LAB, LC2							
	Cyclopentano-naphthalene	N	N	N	N	N	SASS, LAB, LC2							
	2,3-Benzofluorene	N	N	N	N	N	SASS, LAB, LC3							
	1,2-Benzofluorene	N	N	N	N	N	SASS, LAB, LC3							
	Cyclopenta(def)-phenanthrene	N	N	N	N	N	SASS, LAB, LC3							
	Truxene(Tribenzylene Benzene)	N	N	N	N	N	SASS, LAB, LC3							

TABLE KEY:

1. TEST METHOD:
   1. STANDARD
   2. DEVELOPMENTAL
   3. UNKNOWN

   A. AAX
   B. XRD
   C. WET CHEMICAL
   D. ESCA
   E. GC/MS
2. EXPECTED TEST SUCCESS:
   1. HIGH
   2. MODERATE
   3. UNKNOWN
3. TEST COST
   1. REASONABLE
   2. MODERATE
   3. HIGH
4. SAMPLE ALIQUOT
   1. ADEQUATE
   2. MARGINAL
   3. INADEQUATE
   4. RESAMPLE

Level 1 Data Reduction and Decision Charts (Continued)

Level 1 Data Reduction and Decision Charts (Continued)

CATEGORY	COMPOUND	MATE AIR μg/m³ (ppm)	MATE WATER μg/l HEALTH	MATE WATER μg/l ECOLOGY	MATE LAND μg/g HEALTH	MATE LAND μg/g ECOLOGY	WHERE FOUND IN LEVEL I	SAMPLE μg/m³ μg/g μg/l	RATIO SAMPLE/MATE	LEVEL 2 REQUIRED Y=YES N=NO	TEST METHOD[1]	TEST EXPECTATIONS[2]	TEST COST[3]	SAMPLE ALIQUOT[4]
23. HETEROCYCLIC NITROGEN														
A. PYRIDINE & SUBSTITUTED PYRIDINES	Pyridine	1.5×10^4 (5)	2.25×10^5	1.0×10^4	4.5×10^2	2.0×10	SASS, LAB, C8							
	Di & polysubstituted pyridines	2.7×10^4	4.05×10^5	N	8.2×10^2	N	SASS, LAB, C10							
	Picolines	3.56×10^4	5.34×10^5	N	1.1×10^3	N	SASS, LAB, C6							
	Chloropyridine	4.82×10^3	7.23×10^4	N	1.4×10^2	N	SASS, LAB, C9 & C10							
	Collidines	5.9×10^4	1.0×10^6	N	2.1×10^3	N	SASS, LAB, C8 & LC3							
	Monosubstituted Alkyl Pyridines	N	N	N	N	N	SASS, LAB, LC3							
B. FUSED 6-MEMBERED RING	Dibenz(a,h) acridine	2.24×10^2	3.36×10^3	N	6.7	N	SASS, LAB, LC6							
	Dibenz(a,j) acridine	2.47×10^2	3.71×10^3	N	7.4	N	SASS, LAB, LC6							
	Benz(c)acridine	1.1×10^4	1.6×10^5	N	3.2×10^2	N	SASS, LAB, LC6							
	Quinoline; Isoquinoline	1.58×10^4	2.37×10^5	N	4.7×10^2	N	SASS, LAB, LC6							
	Dibenz(c,h) acridine	2.33×10^4	3.5×10^5	N	6.9×10^2	N	SASS, LAB, LC6							
	Methylquinolines	5.54×10^4	8.31×10^5	N	1.7×10^3	N	SASS, LAB, LC6							
	Acridine	9.0×10^4	1.4×10^6	N	2.7×10^3	N	SASS, LAB, LC6							
	Dimethylquinolines	N	N	N	N	N	SASS, LAB, LC6							
	Dihydroacridine	N	N	N	N	N	SASS, LAB, LC6							
	Benzo(c)quinolin	N	N	N	N	N	SASS, LAB, LC6							
	Benzo(f)quinoline	N	N	N	N	N	SASS, LAB, LC6							
	Benzo(h)quinoline	N	N	N	N	N	SASS, LAB, LC6							
	Benzo(a)acridine	N	N	N	N	N	SASS, LAB, LC6							
	Dibenz H-Indeno (1,2-b)quinoline	N	N	N	N	N	SASS, LAB, LC6							
	Indeno (1,2,3,i,j) isoquinoline	N	N	N	N	N	SASS, LAB, LC6							
C. PYRROLE AND RING DERIVATIVES	Dibenzo(c,d) carbazole	1.05×10^3	1.58×10^3	N	3.0	N	SASS, LAB, LC6							
	Pyrrole	2.75×10^3	4.13×10^4	N	8.1×10	N	SASS, LAB, LC6							
	Dibenzo(a,g) carbazole	6.03×10^3	9.05×10^4	N	1.8×10^2	N	SASS, LAB, LC6							
	Indole	1.1×10^4	1.7×10^5	N	3.3×10^2	N	SASS, LAB, LC6							

TABLE KEY:

1. TEST METHOD:
   1. STANDARD
   2. DEVELOPMENTAL
   3. UNKNOWN

   A. AAS
   B. XRD
   C. WET CHEMICAL
   D. ESCA
   E. GC/MS
2. EXPECTED TEST SUCCESS:
   1. HIGH
   2. MODERATE
   3. UNKNOWN
3. TEST COST
   1. REASONABLE
   2. MODERATE
   3. HIGH
4. SAMPLE ALIQUOT
   1. ADEQUATE
   2. MARGINAL
   3. INADEQUATE
   4. RESAMPLE

Level 1 Data Reduction and Decision Charts (Continued)

Level 1 Data Reduction and Decision Charts (Continued)

CATEGORY	COMPOUND	MATE AIR $\mu g/m^3$ (ppm)	MATE WATER $\mu g/l$ HEALTH	MATE WATER $\mu g/l$ ECOLOGY	MATE LAND $\mu g/g$ HEALTH	MATE LAND $\mu g/g$ ECOLOGY	WHERE FOUND IN LEVEL I	SAMPLE $\mu g/m^3$ $\mu g/g$ $\mu g/l$	RATIO $\frac{\text{SAMPLE}}{\text{MATE}}$	LEVEL 2 REQUIRED Y=YES N=NO	TEST METHOD[1]	TEST EXPEC-TATIONS[2]	TEST COST[3]	SAMPLE ALIQUOT[4]
D. NITROGEN HETEROCYCLES containing ADDN'L HETEROATOMS	Dibenzo(a,i) carbazole	1.15×10^4	1.73×10^5	N	3.6×10^2	N	SASS, LAB, LC6							
	Benzo(a) carbazole	1.89×10^4	2.84×10^5	N	5.6×10^2	N	SASS, LAB, LC6							
	Carbazole	2.25×10^4	3.38×10^5	N	6.8×10^2	N	SASS, LAB, LC6							
	Methylindoles	4.5×10^4	6.75×10^5	N	1.4×103	N	SASS, LAB, LC6							
	Benzothiazole	4.28×10^3	6.42×10^4	N	6.4×10^4	N	SASS, LAB, LC6							
	Methyl Benzthiazoles	4.73×10^3	7.10×10^4	N	1.4×10^2	N	SASS, LAB, LC6							
24. HETEROCYCLIC NITROGEN	Tetrahydrofuran	5.9×10^5 (200)	8.85×10^6	N	1.8×10^4	N	SASS, LAB, LC5							
	Furan	N	N	N	N	N	GAS, FIELD, C5							
	Benzofuran	N	N	N	N	N	SASS, LAB, C10							
	Dibenzofuran	N	N	N	N	N	SASS, LAB, LC5							
	Methyldibenzo-furanes	N	N	N	N	N	SASS, LAB, LC5							
	Naphthofurans	N	N	N	N	N	SASS, LAB, LC5							
	Benzo(b)naphto (2,3-d)furan	N	N	N	N	N	SASS, LAB, LC5							
	Phenoanthro(9, 10-b)furan	N	N	N	N	N	SASS, LAB, LC5							
	1-9-Benzox-anthene	N	N	N	N	N	SASS, LAB, LC5							
25. HETEROCYCLIC SULFUR	Benzonaphtho-thiopene	9.86×10^2	1.48×10^4	N	3.0×10	N	SASS, LAB, LC4							
	Thiophene	4.5×10^3	6.75×10^4	N	1.4×10^2	N	GAS, FIELD, C5							
	Methylthiophenes	2.25×10^4	3.38×10^5	N	7.0×10^2	N	SASS, LAB, C8							
	Benzo(b)thiophene	2.30×10^4	3.45×10^5	N	7.0×10^2	N	SASS, LAB, LC4							
	Dimethyl-thiophenes	N	N	N	N	N	SASS, LAB, C9							
	Tri; Tetramethyl thiophenes	N	N	N	N	N	SASS, LAB, C10							
	2,2-Bithiophene	N	N	N	N	N	SASS, LAB, LC4							
	Dibenzthiophene	N	N	N	N	N	SASS, LAB, LC4							
26. ORGANO-METALLICS A. ALKYL or ARYL	Alkyl Mercury	1.0×10 (.001)	1.5×10^2	2.0×10^{-4}	3.0×10^{-1}	4.0×10^{-5}	GAS, FIELD, C6							
	Tetraethyl Lead	1.0×10^2 (.0075)	1.5×10^3	$<4.5 \times 10^1$			SASS, LAB, C12 and LC1							
	Organotin	1.0×10^2	1.0×10^3	N	3.0	N	GAS, FIELD, C6							
	Tetramethyl Lead	1.5×10^2 (.014)	2.25×10^3	N	4.5	N	SASS, LAB, C7							
	Organogermanes	3.15×10^4	4.73×10^5	N	9.4×10^2	N	GAS, FIELD, C6							
	Trimethyl Arsine	N	N	N		N	GAS, FIELD, C5							

TABLE KEY:

1. TEST METHOD:
   1. STANDARD
   2. DEVELOPMENTAL
   3. UNKNOWN

   A. AAS
   B. XRD
   C. WET CHEMICAL
   D. ESCA
   E. GC/MS

2. EXPECTED TEST SUCCESS:
   1. HIGH
   2. MODERATE
   3. UNKNOWN

3. TEST COST
   1. REASONABLE
   2. MODERATE
   3. HIGH

4. SAMPLE ALIQUOT
   1. ADEQUATE
   2. MARGINAL
   3. INADEQUATE
   4. RESAMPLE

Level 1 Data Reduction and Decision Charts (Continued)

Level 1 Data Reduction and Decision Charts (Continued)

CATEGORY	COMPOUND	MATE AIR μg/m³ (ppm)	MATE WATER μg/l HEALTH	MATE WATER μg/l ECOLOGY	MATE LAND μg/g HEALTH	MATE LAND μg/g ECOLOGY	WHERE FOUND IN LEVEL I	SAMPLE μg/m³ μg/g μg/l	RATIO SAMPLE / MATE	LEVEL 2 REQUIRED Y=YES N=NO	TEST METHOD[1]	TEST EXPEC-TATIONS[2]	TEST COST	SAMPLE ALIQUOT[4]
B. SANDWICH TYPE	Nickelocene	3.5×10^3	5.25×10^4	N	1.0×10^2	N	SASS,LAB,LC4							
	Ferrocene	5.94×10^4	8.91×10^5	N	1.8×10^3	N	GAS,FIELD,C6							
	Dibenzene chromium	N	N	N	N	N								
C. METAL PORPHYRINS AND OTHER CHELATES	Complexed Copper	3.52×10^3	5.28×10^4	N	9.0×10	N								
	Complexed Nickel	N	N	N	N	N								
	Complexed Iron	N	N	N	N	N								
	Complexed Tin	N	N	N	N	N								
	Complexed Zinc	N	N	N	N	N								

TABLE KEY:

1. TEST METHOD:
   1. STANDARD
   2. DEVELOPMENTAL
   3. UNKNOWN

   A. AAS
   B. XRD
   C. WET CHEMICAL
   D. ESCA
   E. GC/MS

2. EXPECTED TEST SUCCESS:
   1. HIGH
   2. MODERATE
   3. UNKNOWN

3. TEST COST
   1. REASONABLE
   2. MODERATE
   3. HIGH

4. SAMPLE ALIQUOT
   1. ADEQUATE
   2. MARGINAL
   3. INADEQUATE
   4. RESAMPLE

Level 1 Data Reduction and Decision Charts (Continued)

Level I Data Reduction and Decision Charts (Continued)

CATEGORY	COMPOUND	MATE AIR $\mu g/m^3$ (ppm)	MATE WATER $\mu g/l$ HEALTH	MATE WATER $\mu g/l$ ECOLOGY	MATE LAND $\mu g/g$ HEALTH	MATE LAND $\mu g/g$ ECOLOGY	SAMPLE $\mu g/m^3$ $\mu g/g$ $\mu g/l$	RATIO $\frac{SAMPLE}{MATE}$	LEVEL 2 REQUIRED Y=YES N=NO	TEST METHOD[1]	TEST EXPEC-TATIONS[2]	TEST COST[3]	SAMPLE ALIQUOT[4]
27. LITHIUM	Li	2.2×10	3.3×10^2	3.8×10^2	7.0×10^{-1}	7.5×10^{-1}							
	Li^+	2.2×10	3.3×10^2	3.8×10^2									
	LiF (as Li)	2.2×10	3.3×10^2	3.8×10^2									
	Li_2CO_3 (as Li)	2.2×10	3.3×10^2	3.8×10^2									
	LiH	2.5×10	3.8×10^2	N									
28. SODIUM	Na^+	5.3×10^4	8.0×10^5	N	1.6×10^3	N							
	NaOH	2.0×10^3	3.0×10^4	N	6.0×10	N							
29. POTASSIUM	KOH	2.0×10^3	3.0×10^4	N	6.0×10	N							
	K	N	N	N	N	N							
	K^+ (as K)	N	N	2.3×10^4	N	4.6×10							
30. RUBIDIUM	Rb^{+1}	1.21×10^5	1.82×10^6	N	3.64×10^3	N							
31. CESIUM	CS^{+1}	8.19×10^4	1.23×10^6	N	2.46×10^3	N							
32. BERYLLIUM	Be	2.0	3.0×10	5.5×10	6.0×10^{-2}	1.1×10^{-1}							
	Be^{++}	2.0	3.0×10	5.5×10	6.0×10^{-2}	1.1×10^{-1}							
	BeO (as Be)												
	$BeO \cdot Al_2O_3 \cdot SiO_2$ (as Be)	2.0	3.0×10	5.5×10	6.0×10^{-2}	1.1×10^{-1}							
33. MAGNESIUM	Magnesium, Mg	6.0×10^3	9.0×10^4	8.7×10^4	1.8×10^2	1.7×10^2							
	Magnesium Ion, Mg^{++}	6.01×10^3	9.0×10^4	8.7×10^4	1.8×10^2	1.7×10^2							
	Magnesium Oxide, MgO	1.01×10^4	1.5×10^5	1.0×10^5	3.0×10^2	2.0×10^2							
	Magnesium Fluoride, MgF_2 (as Mg)	6.0×10^3	9.0×10^4	8.7×10^4	1.8×10^2	1.7×10^2							
	Magnesium Sulfate, $MgSO_4$ (as Mg)	6.0×10^3	9.0×10^4	8.7×10^4	1.8×10^2	1.7×10^2							
	Magnesite, $MgCO_3$ (as Mg)	6.0×10^3	9.0×10^4	8.7×10^4	1.8×10^2	1.7×10^2							
	Dolomite, $MgCO_3 \cdot CaCO_3$ (as Mg)	6.0×10^3	9.0×10^4	8.7×10^4	1.8×10^2	1.7×10^2							
	Asbestos (as Mg)	6.0×10^3	9.0×10^4	8.7×10^4	1.8×10^2	1.7×10^2							
34. CALCIUM	Calcium Ion, Ca^{++}	1.6×10^4	2.4×10^5	1.6×10^4	4.8×10^2	3.2×10							
	Calcium Fluoride, CaF_2	N	N	N	N	N							
	Calcium Carbonate, $CaOO_3$	N	N	N	N	N							
	Calcium Sulfate, $CaSO_4$	N	N	N	N	N							
	Dolomite, $MgCO_3 \cdot CaCO_3$	N	N	N	N	N							

TABLE KEY:

1. TEST METHOD
   1. STANDARD
   2. DEVELOPMENTAL
   3. UNKNOWN

   A. AAS
   B. XRD
   C. WET CHEMICAL
   D. ESCA
   E. GC/MS

2. EXPECTED TEST SUCCESS:
   1. HIGH
   2. MODERATE
   3 UNKNOWN

3. TEST COST
   1. REASONABLE
   2. MODERATE
   3. HIGH

4. SAMPLE ALIQUOT
   1. ADEQUATE
   2. MARGINAL
   3. INADEQUATE
   4. RESAMPLE

Level 1 Data Reduction and Decision Charts (Continued)

Level 1 Data Reductionand Decision Charts (Continued)

CATEGORY	COMPOUND	MATE AIR $\mu g/m^3$ (ppm)	MATE WATER $\mu g/l$ HEALTH	MATE WATER $\mu g/l$ ECOLOGY	MATE LAND $\mu g/g$ HEALTH	MATE LAND g/g ECOLOGY	SAMPLE $\mu g/m^3$ $\mu g/g$ $\mu g/l$	RATIO $\frac{SAMPLE}{MATE}$	LEVEL 2 REQUIRED Y=YES N=NO	TEST METHOD[1]	TEST EXPEC-TATIONS[2]	TEST COST[3]	SAMPLE ALIQUOT[4]
35. STRONTIUM	Strontium	3.1×10^3	4.6×10^4	N	9.2×10	N							
	Strontium Ion, Sr^{++} (as Sr)	3.1×10^3	4.6×10^4	N	9.2×10	N							
	Strontium Fluoride, SrF_2 (as Sr)	3.1×10^3	4.6×10^4	N	9.2×10	N							
	Strontium Sulfate, $SrSO_4$ (as Sr)	3.1×10^3	4.6×10^4	N	9.2×10	N							
36. BARIUM	Barium, Ba	5.0×10^2	5.0×10^3	2.5×10^3	1.0×10	5.0							
	Barium Ion, Ba^{++} (as Ba)	5.0×10^2	5.0×10^3	2.5×10^3	1.0×10	5.0							
	Barium Sulfide, BaS (as Ba)	5.0×10^2	5.0×10^3	2.5×10^3	1.0×10	5.0							
	Barium Thiocarbonate, $BaCS_3$ (as Ba)	5.0×10^2	5.0×10^3	2.5×10^3	1.0×10	5.0							
	Barium Fluoride, BaF_2 (as Ba)	5.0×10^2	5.0×10^3	2.5×10^3	1.0×10	5.0							
	Barium Carbonate, $BaCO_3$ (as Ba)	5.0×10^2	5.0×10^3	8.5×10^3	1.0×10	5.0							
	Barium Sulfate, $BaSO_4$ (as Ba)	5.0×10^2	5.0×10^3	2.5×10^3	1.0×10	5.0							
37. BORON	Boron, B	3.1×10^3	4.7×10^4	2.5×10^4	9.3×10	5.0×10							
	Borate, BO_3 (as B)	3.1×10^3	4.7×10^4	2.5×10^4	9.3×10	5.0×10							
	Metaborate, BO_2- (as B)	3.1×10^3	4.7×10^4	2.5×10^4	9.3×10	5.0×10							
	Boron Oxide, B_2O_3	1.0×10^4	1.5×10^5	N	3.0×10^2	N							
38. ALUMINUM	Aluminum, Al	5.2×10^3	8.0×10^4	1.0×10^3	1.6×10^2	2.01							
	Aluminum Ion, Al^{+++}	5.2×10^3	8.0×10^4	1.0×10^3	1.6×10^2	N							
	Bauxite, $Al_2O_3 \cdot 3H_2O$ (as Al)	5.2×10^3	8.0×10^4	1.0×10^3	1.6×10^2	2.0							
	Hydrated Aluminum Silicate (as Al)	5.2×10^3	8.0×10^4	1.0×10^3	1.6×10^2	2.0							
	Alums $[M\,Al\,(SO_4)_2] \cdot (H_2O)_x$ (as Al)	5.2×10^3	8.0×10^4	1.0×10^3	1.6×10^2	2.0							
	Aluminum Oxide, Al_2O_3	1.0×10^4	1.5×10^5	N	3.0×10^2	N							
39. GALLIUM	Gallium, Ga	5.0×10^3	7.4×10^4	N	1.5×10^2	N							
	Elemental Species, Ga	5.0×10^3	7.4×10^4	N	1.5×10^2	N							
	Gallous, Ga^{+1} (as Ga)	5.0×10^3	7.4×10^4	N	1.5×10^2	N							

TABLE KEY:

1. TEST METHOD
   1. STANDARD
   2. DEVELOPMENTAL
   3. UNKNOWN

   A. AAS
   B. XRD
   C. WET CHEMICAL
   D. ESCA
   E. GC/MS
2. EXPECTED TEST SUCCESS:
   1. HIGH
   2. MODERATE
   3. UNKNOWN
3. TEST COST
   1. REASONABLE
   2. MODERATE
   3. HIGH
4. SAMPLE ALIQUOT
   1. ADEQUATE
   2. MARGINAL
   3. INADEQUATE
   4. RESAMPLE

Level 1 Data Reduction and Decision Charts (Continued)

Level 1 Data Reduction and Decision Charts (Continued)

CATEGORY	COMPOUND	MATE AIR $\mu g/m^3$ (ppm)	MATE WATER $\mu g/l$ HEALTH	MATE WATER $\mu g/l$ ECOLOGY	MATE LAND $\mu g/g$ HEALTH	MATE LAND $\mu g/g$ ECOLOGY	SAMPLE $\mu g/m^3$ $\mu g/g$ $\mu g/l$	RATIO $\frac{SAMPLE}{MATE}$	LEVEL 2 REQUIRED Y=YES N=NO	TEST METHOD[1]	TEST EXPEC-TATIONS[2]	TEST COST[3]	SAMPLE ALIQUOT[4]
	Gallic, Ga^{+3} (as Ga)	5.0×10^3	7.4×10^4	N	1.5×10^2	N							
	Gallium Sesqui-oxide, Ga_2O_3 (as Ga)	5.0×10^3	7.4×10^4	N	1.5×10^2	N							
40. INDIUM	Indium, In	1.0×10^2	1.5×10^3	N	3.0	N							
	Indium Ion, In^{+3}	1.0×10^2	1.5×10^3	N	3.0	N							
41. THALLIUM	Thallium, Tl	1.0×10^2	1.5×10^3	N	3.0	N							
	Thallous, Tl^{+1}	1.0×10^2	1.5×10^3	N	3.0	N							
	Thallic, Tl^{+3}	1.0×10^2	1.5×10^3	N	3.0	N							
42. CARBON	Elemental Carbon	3.5×10^3	5.3×10^4	N	1.6×10^2	N							
	Coal	N	N	N	N	N							
	Carbide, C^-	N	N	N	N	N							
	Carbonate, CO_3^{-2}	N	N	N	N	N							
	Bicarbonate, HCO_3	N	N	N	N	N							
	Carbonyl, $CO^=$	N	N	N	N	N							
	Carbon Monoxide	4.0×10^3 (35)	6.0×10^5	6.0×10	N/A	N/A							
	Carbon Dioxide	9.0×10^6 (5000)	N	N	N/A	N/A							
43. SILICON	Silane, SiH_4	7.0×10^2	1.1×10^4	N	N	2.1×10							
	Silicon, Si	1.0×10^4	1.5×10^5	N	3.0×10^2	N							
	Orthosilicate, SiO_4^{-2}	N	N	N	N	N							
	Metasilicate, SiO_3^{-2}	N	N	N	N	N							
	Silicon Dioxide, SiO_2	1.0×10^4	1.5×10^5	N	3.0×10^2	N							
	Silicon Disulfide, SiS_2	N	N	N	N	N							
	Silicon Carbide, SiC	1.0×10^4	1.5×10^5	N	3.0×10^2	N							
44. GERMANIUM	Germanium, Ge	5.6×10^2	8.4×10^3	N	1.7×10	N							
	Germanous, Ge^{+2} (as Ge)	5.6×10^2	8.4×10^3	N	1.7×10	N							
	Germanic, Ge^{+4} (as Ge)	5.6×10^2	8.4×10^3	N	1.7×10	N							
	Germanous Sulfide, GeS (as Ge)	5.6×10^2	8.4×10^3	N	1.7×10	N							
	Germanic Sulfice, GeS_2 (as Ge)	5.6×10^2	8.4×10^3	N	1.7×10	N							
	Germane, GeH_4 (as Ge)	5.6×10^2	8.4×10^3	N	1.7×10	N							
	Germanium Oxide, GeO_2 (as Ge)	5.6×10^2	8.4×10^3	N	1.7×10	N							

TABLE KEY:

1. TEST METHOD
   1. STANDARD
   2. DEVELOP-MENTAL
   3. UNKNOWN

   A. AAS
   B. XRD
   C. WET CHEMICAL
   D. ESCA
   E. GC/MS
2. EXPECTED TEST SUCCESS:
   1. HIGH
   2. MODERATE
   3. UNKNOWN
3. TEST COST
   1. REASONABLE
   2. MODERATE
   3. HIGH
4. SAMPLE ALIQUOT
   1. ADEQUATE
   2. MARGINAL
   3. INADEQUATE
   4. RESAMPLE

Level 1 Data Reduction and Decision Charts (Continued)

Level 1 Data Reduction and Decision Charts (Continued)

CATEGORY	COMPOUND	MATE AIR μg/m^3 (ppm)	MATE WATER μg/l HEALTH	MATE WATER μg/l ECOLOGY	MATE LAND μg/g HEALTH	MATE LAND μg/g ECOLOGY	SAMPLE μg/m^3 μg/g μg/l	RATIO $\frac{SAMPLE}{MATE}$	LEVEL 2 REQUIRED Y=YES N=NO	TEST METHOD[1]	TEST EXPEC-TATIONS[2]	TEST COST[3]	SAMPLE ALIQUOT[4]
45. TIN	Tin Oxide, SnO_2	1.0×10^4	1.5×10^5	N	3.0	N							
	Tin, Sn	N	N	N	N	N							
	Stannous, Sn^{+2}	N	N	N	N	N							
	Stannic, Sn^{+4}	N	N	N	N	N							
46. LEAD	Lead, Pb	1.5×10^2	2.5×10^2	5.0×10	5.0×10^{-1}	1.0×10^{-1}							
	Elemental Led, Pb	1.5×10^2	2.5×10^2	5.0×10	5.0×10^{-1}	1.0×10^{-1}							
	Plumbous, Pb^{+2}	1.5×10^2	2.5×10^2	5.0×10	5.0×10^{-1}	1.0×10^{-1}							
	Plumbic, Pb^{+4} (as Pb)	1.5×10^2	2.5×10^2	5.0×10	5.0×10^{-1}	1.0×10^{-1}							
	Lead Monoxide, PbO (as Pb)	1.5×10^2	2.5×10^2	5.0×10	5.0×10^{-1}	1.0×10^{-1}							
	Lead Sulfate, $PbSO_4$ (as Pb)	1.5×10^2	2.5×10^2	5.0×10	5.0×10^{-1}	1.0×10^{-1}							
	Lead Sulfate, Pbs (as Pb)	1.5×10^2	2.5×10^2	5.0×10	5.0×10^{-1}	1.0×10^{-1}							
	Lead Carbonate, $PbCO_3$ (as Pb)	1.5×10^2	2.5×10^2	5.0×10	5.0×10^{-1}	1.0×10^{-1}							
	Lead Phosphate, $Pb_3(PO_4)_2$ (as Pb)	1.5×10^2	2.5×10^2	5.0×10	5.0×10^{-1}	1.0×10^{-1}							
	Lead Chromate, $PbCrO_4$ (as Pb)	1.5×10^2	2.5×10^2	5.0×10	5.0×10^{-1}	1.0×10^{-1}							
	Lead Molybdate, $PbMoO_4$ (as Pb)	1.5×10^2	2.5×10^2	5.0×10	5.0×10^{-1}	1.0×10^{-1}							
	Lead Arsenate, $PbHAsO_4$ (as Pb)	1.5×10^2	2.5×10^2	5.0×10	5.0×10^{-1}	1.0×10^{-1}							
47. NITROGEN	Hydrazine	1.5×10^2 (.1)	2.3	N	4.5	N							
	Alkali Cyanides, NaCN, KCN	5.0×10^3	5.0×10^2	2.5×10	1.0	5.0×10^{-2}							
	Nitric Acid, HNO_3	5.0×10^3	7.5×10^4	4.5×10^2	1.5×10^3	9.0×10^{-1}							
	Nitrogen Oxides, N_2O, NO_2, N_2O_4, N_2O_3, N_2O_5	9.0×10^3	1.4×10^5	N	N/A	N/A							
	Hydrogen Cyanide, HCN	1.1×10^4 (10)	5.0×10^2	2.5×10	1.0	5.0×10^{-2}							
	Ammonia, NH_3	1.8×10^4 (25)	2.5×10^3	5.0×10	5.0	1.0×10^{-1}							
	Cyanogen, C_2N_2	2.0×10^4	1.0×10^3	2.5×10	2.0	5.0×10^{-2}							
	Nitride, N=	N	N	N	N	N							
	Nitrate, No_3-	N	N	N	N	N							
	Nitrite, No_2-	N	N	N	N	N							
	Ammonium, NH_4^+	N	N	N	N	N							

TABLE KEY:

1. TEST METHOD
   1. STANDARD
   2. DEVELOPMENTAL
   3. UNKNOWN

   A. AAS
   B. XRD
   C. WET CHEMICAL
   D. ESCA
   E. GC/MS

2. EXPECTED TEST SUCCESS
   1. HIGH
   2. MODERATE
   3. UNKNOWN

3. TEST COST
   1. REASONABLE
   2. MODERATE
   3. HIGH

4. SAMPLE ALIQUOT
   1. ADEQUATE
   2. MARGINAL
   3. INADEQUATE
   4. RESAMPLE

Level 1 Data Reduction and Decision Charts (Continued)

Level 1 Data Reduction and Decision Charts (Continued)

CATEGORY	COMPOUND	MATE AIR $\mu g/m^3$ (ppm)	MATE WATER $\mu g/l$ HEALTH	MATE WATER $\mu g/l$ ECOLOGY	MATE LAND $\mu g/g$ HEALTH	MATE LAND $\mu g/g$ ECOLOGY	SAMPLE $\mu g/m^3$ $\mu g/g$ $\mu g/l$	RATIO $\frac{\text{SAMPLE}}{\text{MATE}}$	LEVEL 2 REQUIRED Y=YES N=NO	TEST METHOD[1]	TEST EXPECTATIONS[2]	TEST COST[3]	SAMPLE ALIQUOT[4]
48. PHOSPHORUS	Phosphorus, P	1.0×10^2	1.5×10^4	5.0×10^{-1}	3.0×10	1.0×10^{-3}							
	Phosphite, PO_3^{-3} (as P)	1.0×10^2	1.5×10^4	5.0×10^{-1}	3.0×10	1.0×10^{-3}							
	Biphosphate, $H_2PO_4^-$ (as P)	1.0×10^2	1.5×10^4	5.0×10^{-1}	3.0×10	1.0×10^{-3}							
	Phosphine, PH_3	4.0×10^2 (0.3)	6.0×10^3	N	N/A	N/A							
	Phosphoric Acid, H_3PO_4	1.0×10^3	1.5×10^4	4.5×10^3	3.0×10	9.0							
	Phosphorus Pentasulfide	1.0×10^3	1.5×10^4	N	3.0×10	N							
	Phosphate, PO_4^{-3}	N	N	N	N	N							
49. ARSENIC	Arsenic, As	2.0	2.5×10^2	5.0×10^1	5.0×10^{-1}	1.0×10^{-1}							
	Metallic Arsenic	2.0	2.5×10^2	5.0×10^1	5.0×10^{-1}	1.0×10^{-1}							
	Arsenous, As^{+3}	2.0×10	2.5×10^2	5.0×10^1	5.0×10^{-1}	1.0×10^{-1}							
	Arsenic, As^{+5}	2.0×10	2.5×10^2	5.0×10^1	5.0×10^{-1}	1.0×10^{-1}							
	Arsenate, AsO_4^{-3} (as As)	2.0×10	2.5×10^2	5.0×10^1	5.0×10^{-1}	1.0×10^{-1}							
	Arsenite, AsO_3^{-3} (as As)	2.0×10	2.5×10^2	5.0×10^1	5.0×10^{-1}	1.0×10^{-1}							
	Arsenide, As^{-3} (as As)	2.0×10	2.5×10^2	5.0×10^1	5.0×10^{-1}	1.0×10^{-1}							
	Arsine, AsH_3	2.0	2.5×10^2	5.0×10^1	5.0×10^{-1}	1.0×10^{-1}							
	Arsenic Trioxide, As_2O_3	2.0	2.5×10^2	5.0×10^1	5.0×10^{-1}	1.0×10^{-1}							
50. ANTIMONY	Antimony Trioxide, Sb_2O_3	5.0×10^1	7.5×10^2	2.0×10^2 (as Sb)	1.5	4.0×10^{-1} (as Sb)							
	Antimony Metal, Sb	5.0×10^2	7.5×10^3	2.0×10^2	1.5×10^1	4.0×10^{-1}							
	Antimonous, (stibnous) Sb^{+3}	5.0×10^2	7.5×10^3	2.0×10^2	1.5×10^1	4.0×10^{-1}							
	Antimonic (stibnic) Sb^{+5}	5.0×10^2	7.5×10^3	2.0×10^2	1.5×10^1	4.0×10^{-1}							
	Stibine, SbH_3 (as Sb)	5.0×10^2	7.5×10^3	2.0×10^2	1.5×10^1	4.0×10^{-1}							
	Antimonous Sulfide, Sb_2S_3	5.0×10^2	7.5×10^3	2.0×10^2	1.5×10^1	4.0×10^{-1}							
	Antimony, Sb	5.0×10^2	7.5×10^3	2.0×10^2	1.5×10^1	4.0×10^{-1}							
51. BISMUTH	Bismuth, Bi	4.1×10^2	6.1×10^3	N	1.2×10^1	N							
	Elemental Bismuth, Bi	4.1×10^2	6.1×10^3	N	1.2×10^1	N							
	Bismuthous, Bi^{+3} (as Bi)	4.1×10^2	6.1×10^3	N	1.2×10^1	N							
	Bismuthic, Bi^{+5} (as Bi)	4.1×10^2	6.1×10^3	N	1.2×10^1	N							

TABLE KEY:

1. TEST METHOD
   1. STANDARD
   2. DEVELOPMENTAL
   3. UNKNOWN

   A. AAS
   B. XRD
   C. WET CHEMICAL
   D. ESCA
   E. GS/MS
2. EXPECTED TEST SUCCESS:
   1. HIGH
   2. MODERATE
   3. UNKNOWN
3. TEST COST
   1. REASONABLE
   2. MODERATE
   3. HIGH
4. SAMPLE ALIQUOT
   1. ADEQUATE
   2. MARGINAL
   3. INADEQUATE
   4. RESAMPLE

Level 1 Data Reduction and Decision Charts (Continued)

Level 1 Data Reduction and Decision Charts (Continued)

CATEGORY	COMPOUND	MATE AIR µg/m³ (ppm)	MATE WATER µg/l HEALTH	MATE WATER µg/l ECOLOGY	MATE LAND µg/g HEALTH	MATE LAND µg/g ECOLOGY	SAMPLE µg/m³ µg/g µg/l	RATIO SAMPLE/MATE	LEVEL 2 REQUIRED Y=YES N=NO	TEST METHOD[1]	TEST EXPECTATIONS[2]	TEST COST[3]	SAMPLE ALIQUOT[4]
52. OXYGEN	Ozone, O_3	2.0×10^2 (0.1)	N/A	N/A	N/A	N/A							
53. SULFUR	Rhombic Sulfur, S_8	N	N	N	N	N							
	Sulfide, S^{-2}	N	N	N	N	N							
	Sulfate, SO_4^{-2}	N	N	N	N	N							
	Sulfite, SO_3^{-2}	N	N	N	N	N							
	Thiocyanate, SCN^-	N	N	N	N	N							
	Sulfur Trioxide, SO_3	N	N	N	N	N							
	Sulfuric Acid, H_2SO_4	1.0×10^3	1.5×10^4	4.5×10^2	3.0×10^1	9.0×10^2							
	Sulfur Dioxides, SO_2	1.3×10^4	2.0×10^5	N	4.0×10^2	N							
	Hydrogen Sulfide, H_2S	1.5×10^4 (10)	2.3×10^4	1.0×10^1	N/A	N/A							
	Carbon Disulfide, CS_2	6.0×10^4 (20)	9.0×10^5	1.0×10^4	N/A	N/A							
	Carbonyl Sulfide, COS	4.4×10^5	N/A	N/A	N/A	N/A							
54. SELENIUM	Selenium, Se	2.0×10^2	5.0×10^1	2.5×10^1	1.0×10^{-1}	5.0×10^{-2}							
	Elemental Selenium, Se	2.0×10^2	5.0×10^1	2.5×10^1	1.0×10^{-1}	5.0×10^{-2}							
	Selenide, Se^{-2}	2.0×10^2	5.0×10^1	2.5×10^1	1.0×10^{-1}	5.0×10^{-2}							
	Selenites, SeO_3^{-2} (as Se)	2.0×10^2	5.0×10^1	2.5×10^1	1.0×10^{-1}	5.0×10^{-2}							
	Selenates, SeO_4^{-2} (as Se)	2.0×10^2	5.0×10^1	2.5×10^1	1.0×10^{-1}	5.0×10^{-2}							
	Hydrogen Selenide, H_2Se	2.0×10^2 (.05)	5.0×10^1 (as Se)	2.5×10^1 (as Se)	1.0×10^{-1} (as Se)	5.0×10^{-2} (as Se)							
	Carbon Diselenide, CSe_2 (as Se)	2.0×10^2	5.0×10^1	2.5×10^1	1.0×10^{-1}	5.0×10^{-2}							
	Selenium Dioxide, SeO_2 (as Se)	2.0×10^2	5.0×10^1	2.5×10^1	1.0×10^{-1}	5.0×10^{-2}							
55. TELLURIUM	Tellurium, Te	1.0×10^2	1.5×10^3	N	3.0	N							
	Telluride, Te^{-2}	1.0×10^2	1.5×10^3	N	3.0	N							
	Tellurite, TeO_3^{-2} (as Te)	1.0×10^2	1.5×10^3	N	3.0	N							
	Tellurate, TeO_4 (as Te)	1.0×10^2	1.5×10^3	N	3.0	N							
56. FLUORIDE	Fluoride Ion, F^-	2.5×10^3	3.8×10^4	N	7.5×10^1	N							
	Hydrogen Fluoride, HF	2.0×10^3	3.0×10^4	N	N/A	N/							

TABLE KEY

1. TEST METHOD
   1. STANDARD
   2. DEVELOPMENTAL
   3. UNKNOWN

   A. AAS
   B. XRD
   C. WET CHEMICAL
   D. ESCA
   E. GS/MS

2. EXPECTED TEST SUCCESS
   1. HIGH
   2. MODERATE
   3. UNKNOWN

3. TEST COST
   1. REASONABLE
   2. MODERATE
   3. HIGH

4. SAMPLE ALIQUOT
   1. ADEQUATE
   2. MARGINAL
   3. INADEQUATE
   4. RESAMPLE

Level 1 Data Reduction and Decision Charts (Continued)

Level 1 Data Reduction and Decision Charts (Continued)

CATEGORY	COMPOUND	MATE AIR µg/m^3 (ppm)	MATE WATER µg/l HEALTH	MATE WATER g/l ECOLOGY	MATE LAND µg/g HEALTH	MATE LAND µg/g ECOLOGY	SAMPLE µg/m^3 µg/g µg/l	RATIO SAMPLE/MATE	LEVEL 2 REQUIRED Y=YES N=NO	TEST METHOD[1]	TEST EXPECTATIONS[2]	TEST COST[3]	SAMPLE ALIQUOT[4]
57. CHLORINE	Chloride Ion, Cl^-	N	1.3×10^{6}	N	2.6×10^{3}	N							
	Hypochlorite, ClO^-	N	N	N	N	N							
	Chlorite, ClO_2^-	N	N	N	N	N							
	Chlorate, ClO_3	N	N	N	N	N							
	Chlorine Dioxide, ClO_2	N	N	N	N	N							
	Carbonyl Chloride (phosgene), $COCl_2$	4.0×10^{2}	6.0×10^{3}	N	N/A	N/A							
	Hydrogen Chloride, HCl	7.0×10^{3}	1.1×10^{5}	N	N/A	N/A							
58. BROMINE	Bromide Ion, Br^-	N	N	N	N	N							
	Bromide Ion, Br^-	N	N	N	N	N							
	Hydrogen Bromide, HBr	1.0×10^{4}	1.5×10^{5}	N	N/A	N/A							
59. IODINE	Iodide Ion, I^-	N	N	N	N	N							
60. SCANDIUM	Scandium, Sc	5.3×10^{4}	8.0×10^{5}	N	1.6×10^{-3}	N							
	Scandium Ion, Sc^{+3}	5.3×10^{4}	8.0×10^{5}	N	1.6×10^{-3}	N							
61. YTTRIUM	Yttrium Ion, Y^{+3}	1.0×10^{3}	1.5×10^{4}	N	3.0×10^{1}	N							
62. TITANIUM	Titanium, Ti	6.0×10^{3}	9.0×10^{4}	8.2×10^{2} (as $Ti[SO_4]_2$)	1.8×10^{2}	1.6							
	Titanous, Ti^{+3} (as Ti)	6.0×10^{3}	9.0×10^{4}	8.2×10^{2}	1.8×10^{2}	1.6×10							
	Titanic, Ti^{+4} (as Ti)	6.0×10^{3}	9.0×10^{4}	8.2×10^{2}	1.8×10^{2}	1.6×10							
	Titanium Dioxide, TiO_2 (as Ti)	6.0×10^{3}	9.0×10^{4}	8.2×10^{2}	1.8×10^{2}	1.6×10							
63. ZIRCONIUM	Zirconium Ion, Zr^{+4}	5.0×10^{3}	7.5×10^{4}	N	1.5×10^{1}	N							
	Zirconium Dioxide, ZrO_2 (as Zr)	5.0×10^{3}	7.5×10^{4}	N	1.5×10^{1}	N							
64. HAFNIUM	Hafnium Ion, Hf^{+4}	5.0×10^{2}	7.5×10^{4}	N	1.5×10	N							
65. VANADIUM	Vanadium, V	5.0×10^{2}	2.5×10^{3}	1.5×10^{2}	5.0×10	3.0×10^{-1}							
	Elemental Vanadium, V	5.0×10^{2}	2.5×10^{3}	1.5×10^{2}	5.0×10	3.0×10^{-1}							
	Vanadic, V^{+3} (as V)	5.0×10^{2}	2.5×10^{3}	1.5×10^{2}	5.0×10	3.0×10^{-1}							
	Vanadyl, Vo^{+2} (as V)	5.0×10^{2}	2.5×10^{3}	1.5×10^{2}	5.0×10	3.0×10^{-1}							
	Orthovanadate, Vo_4^{-4} (as V)	5.0×10^{2}	2.5×10^{3}	1.5×10^{2}	5.0×10	3.0×10^{-1}							

TABLE KEY:

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   3. UNKNOWN

   A. AAS
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   C. WET CHEMICAL
   D. ESCA
   E. GC/MS
2. EXPECTED TEST SUCCESS:
   1. HIGH
   2. MODERATE
   3. UNKNOWN
3. TEST COST
   1. REASONABLE
   2. MODERATE
   3. HIGH
4. SAMPLE ALIQUOT
   1. ADEQUATE
   2. MARGINAL
   3. INADEQUATE
   4. RESAMPLE

Level 1 Data Reduction and Decision Charts (Continued)

Level 1 Data Reduction and Decision Charts (Continued)

CATEGORY	COMPOUND	MATE AIR $\mu g/m^3$ (ppm)	MATE WATER $\mu g/l$ HEALTH	MATE WATER $\mu g/l$ ECOLOGY	MATE LAND $\mu g/g$ HEALTH	MATE LAND $\mu g/g$ ECOLOGY	SAMPLE $\mu g/m^3$ $\mu g/g$ $\mu g/l$	RATIO $\frac{\text{SAMPLE}}{\text{MATE}}$	LEVEL 2 REQUIRED Y=YES N=NO	TEST METHOD[1]	TEST EXPEC-TATIONS[2]	TEST COST[3]	SAMPLE ALIQUOT[4]
	Metavanadate, VO_3^- (as V)	5.0×10^2	2.5×10^3	1.5×10^2	5.0	3.0×10^{-1}							
	Vanadylic, VO^{+3} (as V)	5.0×10^2	2.5×10^3	1.5×10^2	5.0	3.0×10^{-1}							
	Vanadium Monoxide, VO (as V)	5.0×10^2	2.5×10^3	1.5×10^2	5.0	3.0×10^{-1}							
	Vanadium Trioxide, V_2O_3 (as V)	5.0×10^2	2.5×10^3	1.5×10^2	5.0	3.0×10^{-1}							
	Vanadium Tetraoxide, V_2O_4, (as V)	5.0×10^2	2.5×10^3	1.5×10^2	5.0	3.0×10^{-1}							
	Vanadium Pentoxide, V_2O_5 (as V)	5.0×10^2	2.5×10^3	1.5×10^2	5.0	3.0×10^{-1}							
	Vanadium Carbide, VC (as V)	5.0×10^2	2.5×10^3	1.5×10^2	5.0	3.0×10^{-1}							
	Vanadium Monosulfide, VS (as V)	5.0×10^2	2.5×10^3	1.5×10^2	5.0	3.0×10^{-1}							
	Vanadium Nitride, VN (as V)	5.0×10^2	2.5×10^3	1.5×10^2	5.0	3.0×10^{-1}							
	Vanadyl Sulfate, $VOSO_4 \cdot 5H_2O$ (as V)	5.0×10^2	2.5×10^3	1.5×10^2	5.0	3.0×10^{-1}							
66. NIOBIUM	Niobus, Nb^{+3}	2.2×10^4	3.3×10^5	N	6.5×10^2	N							
	Niobic, Nb^{+5}	2.2×10^4	3.3×10^5	N	6.5×10^2	N							
	Niobium Oxides, NbO, Nb_2O_5 (as Nb)	2.2×10^4	3.3×10^3	N	6.5×10^2	N							
67. TANTALUM	Tantalum Ion, Ta^{+5}	5.0×10^3	7.5×10^4	N	1.5×10^2	N							
68. CHROMIUM	Chromium, Cr	1.0	2.5×10^2	2.5×10^2	5.0×10^{-1}	5.0×10^{-1}							
	Chromous, Cr^{+2} (as Cr)	1.0	2.5×10^2	2.5×10^2	5.0×10^{-1}	5.0×10^{-1}							
	Chromic, Cr^{+3} (as Cr)	1.0	2.5×10^2	2.5×10^2	5.0×10^{-1}	5.0×10^{-1}							
	Chromates, CrO_4^{-2} (as Cr)	1.0	2.5×10^2	2.5×10^2	5.0×10^{-1}	5.0×10^{-1}							
	Chromites, $Cr_2O_4^{-2}$ (as Cr)	1.0	2.5×10^2	2.5×10^2	5.0×10^{-1}	5.0×10^{-1}							
	Dichromates, $Cr_2O_7^{-2}$ (as Cr)	1.0	2.5×10^2	2.5×10^2	5.0×10^{-1}	5.0×10^{-1}							
	Chromium Carbonyl, $Cr(CO)_6$ (as Cr)	1.0	2.5×10^2	2.5×10^2	5.0×10^{-1}	5.0×10^{-1}							
	Chromium Sulfide, Cr_2S_3	1.0	2.5×10^2	2.5×10^2	5.0×10^{-1}	5.0×10^{-1}							
	Chromic Oxide, Cr_2O_3 (as Cr)	1.0	2.5×10^2	2.5×10^2	5.0×10^{-1}	5.0×10^{-1}							
	Chromite Mineral, FeO Cr_2O_3 (as Cr)	1.0	2.5×10^2	2.5×10^2	5.0×10^{-1}	5.0×10^{-1}							

TABLE KEY

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   3. UNKNOWN

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   D. ESCA
   E. GC/MS
2. EXPECTED TEST SUCCESS:
   1. HIGH
   2. MODERATE
   3. UNKNOWN
3. TEST COST
   1. REASONABLE
   2. MODERATE
   3. HIGH
4. SAMPLE ALIQUOT
   1. ADEQUATE
   2. MARGINAL
   3. INADEQUATE
   4. RESAMPLE

Level 1 Data Reduction and Decision Charts (Continued)

Level 1 Data Reduction and Decision Charts (Continued)

CATEGORY	COMPOUND	MATE AIR μg/m^3 (ppm)	MATE WATER μg/l HEALTH	MATE WATER μg/l ECOLOGY	MATE LAND μg/g HEALTH	MATE LAND μg/g ECOLOGY	SAMPLE μg/m^3 μg/g μg/l	RATIO $\frac{\text{SAMPLE}}{\text{MATE}}$	LEVEL 2 REQUIRED Y=YES N=NO	TEST METHOD[1]	TEST EXPEC-TATIONS[2]	TEST COST[3]	SAMPLE ALIQUOT[4]
	Hydrous Chromium Phosphate, $CrPO_4$ XH_2O (as Cr)	1.0	2.5×10^2	2.5×10^2	5.0×10^{-1}	5.0×10^{-1}							
	Iron Chromate, $FeCrO_4$ (as Cr)	1.0	2.5×10^2	2.5×10^2	5.0×10^{-1}	5.0×10^{-1}							
69. MOLYBDEN	Molybdenum, Mo	5.0×10^3	7.5×10^4	7.0×10^3	1.5×10^2	1.4×10^1							
	Molybdenous, Mo^{+2}	5.0×10^3	7.5×10^4	7.0×10^3	1.5×10^2	1.4×10^1							
	Molybdic, Mo^{+3}	5.0×10^3	7.5×10^4	7.0×10^3	1.5×10^2	1.4×10^1							
	Molybdate, MoO_4^{-2} (as Mo)	5.0×10^3	7.5×10^4	7.0×10^3	1.5×10^2	1.4×10^1							
	Molybdenum Sulfide, MoS_2 (as Mo)	5.0×10^3	7.5×10^4	7.0×10^3	1.5×10^2	1.4×10^1							
	Molybdenum Trioxide, MoO_3 (as Mo)	5.0×10^3	7.5×10^4	7.0×10^3	1.5×10^2	1.4×10^1							
70. TUNGSTEN	Tungsten, W	1.0×10^3	1.5×10^4	N	3.0×10^1	N							
	Tungsten Ions, W^{+2}, W^{+4}, W^{+5}, W^{+6}, WO_4^{-2}	N	N	N	N	N							
	Tungsten Disulfide, WS_2 (as W)	1.0×10^3	1.5×10^4	N	3.0×10^1	N							
	Wolframite Mineral, $FeWO_4 \cdot MnWO_4$ (as W)	1.0×10^3	1.5×10^4	N	3.0×10^1	N							
71. MANGANESE	Manganese, Mn	5.0×10^3	2.5×10^2	1.0×10^2	5.0×10^{-1}	2.0×10^{-1}							
	Manganous, Mn^{+2}	5.0×10^3	2.5×10^2	1.0×10^2	5.0×10^{-1}	2.0×10^{-1}							
	Manganic, Mn^{+3}	5.0×10^3	2.5×10^2	1.0×10^2	5.0×10^{-1}	2.0×10^{-1}							
	Permanganate, MnO_4^- (as Mn)	5.0×10^3	2.5×10^2	1.0×10^2	5.0×10^{-1}	2.0×10^{-1}							
	Manganous Oxide, MnO (as Mn)	5.0×10^3	2.5×10^2	1.0×10^2	5.0×10^{-1}	2.0×10^{-1}							
	Manganese Dioxide, MnO_2 (as Mn)	5.0×10^3	2.5×10^2	1.0×10^2	5.0×10^{-1}	2.0×10^{-1}							
	Manganese Carbonate, $McCO_3$ (as Mn)	5.0×10^3	2.5×10^2	1.0×10^2	5.0×10^{-1}	2.0×10^{-1}							
	Manganous Sulfate, $MnSO_4$ (as Mn)	5.0×10^3	2.5×10^2	1.0×10^2	5.0×10^{-1}	2.0×10^{-1}							
	Manganese Sulfide, MnS_2 (as Mn)	5.0×10^3	2.5×10^2	1.0×10^2	5.0×10^{-1}	2.0×10^{-1}							
72. IRON	Iron Carbonyls, $Fe(CO)_5$, $Fe(CO)_9$, $FE_3(CO)_{12}$	7.0×10^2	1.1×10^4	N	2.1×10^1	N							
	Ferrous, Fe^{+2}	1.0×10^3	1.5×10^3	2.5×10^2	3.0×10	5.0×10^{-1}							
	Ferric, Fe^{+3}	1.0×10^3	1.5×10^3	2.5×10^2	3.0×10	5.0×10^{-1}							
	Ferrous Oxide, FeO	5.0×10^3	7.5×10^4	N	1.5×10^2	N							

TABLE KEY:

1. TEST METHOD
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   3. UNKNOWN

   A. AAS
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   D. ESCA
   E. GC/MS
2. EXPECTED TEST SUCCESS:
   1. HIGH
   2. MODERATE
   3. UNKNOWN
3. TEST COST
   1. REASONABLE
   2. MODERATE
   3. HIGH
4. SAMPLE ALIQUOT
   1. ADEQUATE
   2. MARGINAL
   3. INADEQUATE
   4. RESAMPLE

Level 1 Data Reduction and Decision Charts (Continued)

Level 1 Data Reduction and Decision Charts (Continued)

CATEGORY	COMPOUND	MATE AIR $\mu g/m^3$ (ppm)	MATE WATER $\mu g/l$ HEALTH	MATE WATER $\mu g/l$ ECOLOGY	MATE LAND $\mu g/g$ HEALTH	MATE LAND $\mu g/g$ ECOLOGY	SAMPLE $\mu g/m^3$ $\mu g/g$ $\mu g/l$	RATIO $\frac{\text{SAMPLE}}{\text{MATE}}$	LEVEL 2 REQUIRED Y=YES N=NO	TEST METHOD[1]	TEST EXPECTATIONS[2]	TEST COST[3]	SAMPLE ALIQUOT[4]
	Magnetite, $FeO \cdot Fe_2O_3$	9.3×10^3	6.2×10^3	N	3.8×10^1	N							
	Ferrocyanide, $Fe(CN)_6^{-4}$	N	N	N	N	N							
	Ferricyanide, $Fe(CN)_6^{-3}$	N	N	N	N	N							
	Ferric Oxide, Fe_2O_3	N	N	N	N	N							
	Ferric Hydroxide (hydrated) $Fe_2O_3 \cdot XH_2O$	N	N	N	N	N							
	Iron Sulfides, FeS, Fe_2S_3	N	N	N	N	N							
	Pyrite, FeS_2	N	N	$>1.0 \times 10^5$	N	2.0×10^2							
	Potassium Iron Silicate, $KFeSi_2O_6$	N	N	N	N	N							
73. RUTHENIUM	Ruthenium Ion, Ru^{+3}	N	N	N	N	N							
74. COBALT	Cobalt, Co	5.0×10^1	7.5×10^2	2.5×10^2	1.5	5.0×10^{-1}							
	Cobaltous, Co^{+2}	5.0×10^1	7.5×10^2	2.5×10^2	1.5	5.0×10^{-1}							
	Cobaltic, Co^{+3}	5.0×10^1	7.5×10^2	2.5×10^2	1.5	5.0×10^{-1}							
	Cobaltous Carbonate, hydrated, $CoCO_3 \cdot H_2O$ (as Co)	5.0×10^1	7.5×10^2	2.5×10^2	1.5	5.0×10^{-1}							
	Cobalt Carbide, Co_3C (as Co)	5.0×10^1	7.5×10^2	2.5×10^2	1.5	5.0×10^{-1}							
	Cobalt Sulfides, CoS, Co_2S_3 (as Co)	5.0×10^1	7.5×10^2	2.5×10^2	1.5	5.0×10^{-1}							
	Cobalt Arsenic Sulfide, CoAsS (as Co)	5.0×10^1	7.5×10^2	2.5×10^2	1.5	5.0×10^{-1}							
	Cobalt Arsenide, $CoAs_2$ (as Co)	5.0×10^1	7.5×10^2	2.5×10^2	1.5	5.0×10^{-1}							
	Cobalt Carbonyl, $Co(CO)_4$ (as Co)	5.0×10^1	7.5×10^2	2.6×10^2	1.5	5.0×10^{-1}							
	Cobaltous Oxide, CoO (as Co)	5.0×10^1	7.5×10^2	2.5×10^2	1.5	5.0×10^{-1}							
	Cobaltous Hydroxide, $Co(OH)_2$ (as Co)	5.0×10^1	7.5×10^2	2.5×10^2	1.5	5.0×10^{-1}							
75. RHODIUM	Rhodium Ion, Rh^{+3}	1.0×10	1.5×10^1	N	3.0×10^{-2}	N							
76. NICKEL	Nickelous, Ni^{+2}	1.5×10^1	2.3×10^2	1.0×10^1	4.5×10^{-1}	2.0×10^{-2}							
	Nickelic, Ni^{+3}	1.5×10^1	2.3×10^2	1.0×10^1	4.5×10^{-1}	2.0×10^{-2}							
	Nickelous Sulfide, NiS (as Ni)	1.5×10^1	2.3×10^2	1.0×10^1	4.5×10^{-1}	2.0×10^{-2}							
	Nickel Arsenide, NiAs (as Ni)	1.5×10^1	2.3×10^2	1.0×10^1	4.5×10^{-1}	2.0×10^{-2}							

TABLE KEY

1. TEST METHOD
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   E. GC/MS
2. EXPECTED TEST SUCCESS:
   1. HIGH
   2. MODERATE
   3. UNKNOWN
3. TEST COST
   1. REASONABLE
   2. MODERATE
   3. HIGH
4. SAMPLE ALIQUOT
   1. ADEQUATE
   2. MARGINAL
   3. INADEQUATE
   4. RESAMPLE

Level 1 Data Reduction and Decision Charts (Continued)

Level 1 Data Reduction and Decision Charts (Continued)

CATEGORY	COMPOUND	MATE AIR μg/m³ (ppm)	MATE WATER μg/l HEALTH	MATE WATER μg/l ECOLOGY	MATE LAND μg/g HEALTH	MATE LAND μg/g ECOLOGY	SAMPLE μg/m³ μg/g μg/l	RATIO SAMPLE/MATE	LEVEL 2 REQUIRED Y=YES N=NO	TEST METHOD[1]	TEST EXPECTATIONS[2]	TEST COST[3]	SAMPLE ALIQUOT[4]
	Nickel Oxide, NiO (as Ni)	1.5×10^1	2.3×10^2	1.0×10^1	4.5×10^{-1}	2.0×10^{-2}							
	Nickel Antimonide, NiSb (as Ni)	1.5×10^1	2.3×10^2	1.0×10^1	4.5×10^{-1}	2.0×10^{-2}							
	Nickel Arsenic Sulfide, NiAsS (as Ni)	1.5×10^1	2.3×10^2	1.0×10^1	4.5×10^{-1}	2.0×10^{-2}							
	Nickel Carbonyl, $Ni(CO)_4$	4.3×10^1	6.5×10^2	1.0×10^1 (as Ni)	1.0×10^{-1}	2.0×20^{-2}							
77. PLATINUM	Elemental Platinum, Pt	2.0×10	3.0×10^1	N	6.0×10^{-2}	N							
78. COPPER	Copper	2.0×10^2	5.0×10^3	5.0×10^1	1.0×10^1	1.0×10^{-1}							
	Cuprous, Cu^+	2.0×10^2	5.0×10^3	5.0×10^1	1.0×10^1	1.0×10^{-1}							
	Cupric, Cu^{+2}	2.0×10^2	5.0×10^3	5.0×10^1	1.0×10^1	1.0×10^{-1}							
	Copper Fluoride, CuF_2 (as Cu)	2.0×10^2	5.0×10^3	5.0×10^1	1.0×10^1	1.0×10^{-1}							
	Copper Oxides, CuO, Cu_2O (as Cu)	2.0×10^2	5.0×10^3	5.0×10^1	1.0×10^1	1.0×10^{-5}							
	Copper Sulfate, $CuSO_4$ (as Cu)	2.0×10^2	5.0×10^3	5.0×10^1	1.0×10^1	1.0×10^{-1}							
	Copper Sulfides, CuS, Cu_2S (as Cu)	2.0×10^2	5.0×10^3	5.0×10^1	1.0×10^1	1.0×10^{-1}							
	Copper Carbonate, $CuCO_3$ (as Cu)	2.0×10^2	5.0×10^3	5.0×10^1	1.0×10^1	1.0×10^{-1}							
	Malachite Mineral, $CuCO_3 \cdot Cu(OH)_2$ (as Cu)	2.0×10^2	5.0×10^3	5.0×10^1	1.0×10^1	1.0×10^{-1}							
	Chalcopyrite Mineral, $CuFeS_2$	N	N	N	N	N							
79. SILVER	Silver, Ag	1.0×10^1	2.5×10^2	5.0	5.0×10^{-1}	1.0×10^{-2}							
	Silver Ion, Ag^+ (as Ag)	1.0×10^1	2.5×10^2	5.0	5.0×10^{-1}	1.0×10^{-2}							
	Silver Chloride, AgCl (as Ag)	1.0×10^1	2.5×10^2	5.0	5.0×10^{-1}	1.0×10^{-2}							
	Silver Cyanide, AgCN (as Ag)	1.0×10^1	2.5×10^2	5.0	5.0×10^{-1}	1.0×10^{-2}							
	Silver Sulfide, Ag_2S (as Ag)	1.0×10^1	2.5×10^2	5.0	5.0×10^{-1}	1.0×10^{-2}							
80. GOLD	Elemental Gold	N	N	N	N	N							
81. ZINC	Zinc, Zn	4.0×10^3	2.5×10^4	1.0×10^2	5.0×10^1	2.0×10^{-1}							
	Elemental Zinc, Zn	4.0×10^3	2.5×10^4	1.0×10^2	5.0×10^1	2.0×10^{-1}							
	Zinc Ion, Zn^{+2}	4.0×10^3	2.5×10^4	1.0×10^2	5.0×10^1	2.0×10^{-1}							
	Zinc Oxide, ZnO (as Zn)	4.0×10^3	2.5×10^4	1.0×10^2	5.0×10^1	2.0×10^{-1}							

TABLE KEY:

1. TEST METHOD
   1. STANDARD
   2. DEVELOPMENTAL
   3. UNKNOWN

   A. AAS
   B. XRD
   C. WET CHEMICAL
   D. ESCA
   E. GC/MS
2. EXPECTED TEST SUCCESS:
   1. HIGH
   2. MODERATE
   3. UNKNOWN
3. TEST COST
   1. REASONABLE
   2. MODERATE
   3. HIGH
4. SAMPLE ALIQUOT
   1. ADEQUATE
   2. MARGINAL
   3. INADEQUATE
   4. RESAMPLE

Level 1 Data Reduction and Decision Charts (Continued)

Level 1 Data Reduction and Decision Charts (Continued)

CATEGORY	COMPOUND	MATE AIR μg/m³ (ppm)	MATE WATER μg/l HEALTH	MATE WATER μg/l ECOLOGY	MATE LAND μg/g HEALTH	MATE LAND μg/g ECOLOGY	SAMPLE μg/m³ μg/g μg/l	RATIO SAMPLE/MATE	LEVEL 2 REQUIRED Y=YES N=NO	TEST METHOD[1]	TEST EXPEC-TATIONS[2]	TEST COST[3]	SAMPLE ALIQUOT[4]
	Zinc Sulfate, $ZnSO_4$ (as Zn)	4.0×10^3	2.5×10^4	1.0×10^2	5.0×10^1	2.0×10^{-1}							
	Zinc Sulfide, ZnS (as Zn)	4.0×10^3	2.5×10^4	1.0×10^2	5.0×10^1	2.0×10^{-1}							
82. CADMIUM	Cadmium, Cd	1.0×10^1	5.0×10^1	1.0	1.0×10^{-1}	2.0×10^{-3}							
	Elemental Cadmium, Cd	1.0×10^1	5.0×10^1	1.0	1.0×10^{-1}	2.0×10^{-3}							
	Cadmium Ion, Cd^{+2}	1.0×10^1	5.0×10^1	1.0	1.0×10^{-1}	2.0×10^{-3}							
	Cadmium Sulfide, CdS (as Cd)	1.0×10^1	5.0×10^1	1.0	1.0×10^{-1}	2.0×10^{-3}							
	Cadmium Oxide, CdO (as Cd)	1.0×10^1	5.0×10^1	1.0	1.0×10^{-1}	2.0×10^{-3}							
83. MERCURY	Mercury, Hg	5.0×10^1	1.0×10^1	2.5×10^2	2.0×10^{-2}	5.0×10^{-1}							
	Elemental Mercury, Hg	5.0×10^1	1.0×10^1	2.5×10^2	2.0×10^{-2}	5.0×10^{-1}							
	Mercurous, Hg_2^{++}	5.0×10^1	1.0×10^1	2.5×10^2	2.0×10^{-2}	5.0×10^{-1}							
	Mercuric, Hg^{++}	5.0×10^1	1.0×10^1	2.5×10^2	2.0×10^{-2}	5.0×10^{-1}							
	Mercuric Sulfide, HgS	5.0×10^1	1.0×10^1	2.5×10^2	2.0×10^{-2}	5.0×10^{-1}							
	Mercuric Chloride, $HgCl_2$	5.0×10^1	1.0×10^1	2.5×10^2	2.0×10^{-2}	5.0×10^{-1}							
84. CERIUM	Dysprosium, Dy (Dy^{+3})	9.3×10^3	2.3×10^5	N	4.6×10^2	N							
	Cerium, Ce (Ce^{+3}, Ce^{+4}, Ce_2O_3)	3.7×10^4	5.5×10^5	N	1.1×10^3	N							
	Praseodymium, Pr (Pr^{+3})	5.1×10^4	7.7×10^5	N	1.5×10^3	N							
	Samarium, Sm (Sm^{+3})	5.3×10^4	7.9×10^5	N	1.6×10^3	N							
	Lanthanum, La	1.1×10^5	1.7×10^6	N	3.4×10^3	N							
	Neodymium, Nd (Nd^{+3})	N	N	N	N	N							
85. URANIUM	Uranium, U (U^{+6})	9.0×10	1.0×10^4	5.0×10^2	1.2×10^2	1.0							
86. THORIUM	Thorium, Th (Th^{+4})	4.2×10^2	6.3×10^3	N	1.3×10	N							

TABLE KEY:

1. TEST METHOD
   1. STANDARD
   2. DEVELOPMENTAL
   3. UNKNOWN

   A. AAS
   B. XRD
   C. WET CHEMICAL
   D. ESCA
   E. GC/MS

2. EXPECTED TEST SUCCESS:
   1. HIGH
   2. MODERATE
   3. UNKNOWN

3. TEST COST
   1. REASONABLE
   2. MODERATE
   3. HIGH

4. SAMPLE ALIQUOT
   1. ADEQUATE
   2. MARGINAL
   3. INADEQUATE
   4. RESAMPLE

APPENDIX B

LIQUID CHROMATOGRAPHY SEPARATION PROCEDURE

Column: 200 mm x 10.5 mm ID, glass with Teflon stopcock.

Adsorbent: Divison Silica Gel, 60-200 mesh, Grade 950 (Fisher Scientific Company). This adsorbent is activated at 100°C for two hours just prior to use. Cool in a desiccator.

B.1 PROCEDURE FOR COLUMN PREPARATION

Dry pack the chromatographic column, plugged at one end with glass wool, with 6.0 grams of freshly activated silica gel. A portion of properly activated silica gel weighing 6.0 ±0.2 g occupies 8 ml in a 10 ml graduated cylinder. Vibrate the column for a minute to compact the gel bed. Pour pentane into the solvent reservoir positioned above the column and let the pentane flow into the silica gel bed until the column is homogeneous throughout and free of any cracks and trapped air bubbles.* The total height of the silica bed in this packed column is 10 cm. The solvent void volume of the column is 2 to 4 ml. When the column is fully prepared, allow the pentane level in the column to drop to the top of the silica bed so that the sample can be loaded for subsequent chromatographic elution.

B.2 PREPARATION OF THE SAMPLE

At room temperature evaporate the solvent from an aliquot of solution containing the sample. The preferred sample weight is 100 mg. Weigh this sample in a glass weighing funnel. In order to facilitate transfer of the sample, add 0.5 to 1.0 g of activated silica gel to the sample and carefully mix this with the sample using a micro-spatula.

Table B-1 shows the sequence for the chromatographic elution. In order to ensure adequate resolution and reproducibility, the column elution rate is maintained at 1 ml per minute.

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*A convenient device for the elimination of gel bed cracks and air bubbles is acetone coolant, which is subsequently referred to as the ACE B method. It consists of a paper towel wound loosely around the glass column along the region of the crack or bubble; the paper towel is periodically moistened with acetone. The acetone evaporation cools the region and dissipates the bubble or crack.

Table B-1. Liquid Chromatography Elution Sequence

No. Fraction	Solvent Composition	Volume Collected
1	Pentane	25 ml
2	20% Methylene chloride in pentane	10 ml
3	50% Methylene chloride in pentane	10 ml
4	Methylene chloride	10 ml
5	5% Methanol in methylene chloride	10 ml
6	20% Methanol in methylene chloride	10 ml
7	50% Methanol in methylene chloride	10 ml
8	Conc. HCl/Methanol/Methylene chloride (5 + 70 + 30)	10 ml

B.3 LOADING SAMPLE ON THE COLUMN

Quantitatively transfer the sample into the column via the weighing funnel used for sample preparation; a micro-spatula can be used to aid in the sample transfer. Rinse the funnel* with a few ml of pentane to complete the quantitative sample transfer. (Note: Do not rinse with methylene chloride because this solvent will cause the aromatics to elute with the paraffins.) Add the solvent slowly to minimize disturbing the gel bed and eliminate the trapped air bubbles, particularly in the zone of the sample-containing silica gel, by using the ACE B approach (see footnote, preceding page). The chromatographic system is now ready for sample fractionation.

B.4 CHROMATOGRAPHIC SEPARATION INTO 8 FRACTIONS

The volume of solvents shown in Table B-1 represents the solvent volume collected for that fraction. If the volume of solvent collected is less than volume actually added due to evaporation, add solvent as necessary. In all cases, however, the solvent level in the column should be at the end of the collection of any sample fraction.

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*Save this weighing funnel for subsequent additional rinsing with the solvents used at interim fractions up to methylene chloride.

After the first fraction is collected, rinse the original sample weighing funnel with a few ml of the fraction number 2 solvent (20% methylene chloride/pentane) and carefully transfer this rinsing into the column. Repeat as necessary for fractions 3 and 4.

B.5 PREPARATION OF XAD-2 RESIN

The XAD-2 resin to be used in the SASS train sorbent trap must be cleaned prior to use. The resin as obtained from Rohm and Haas is soaked with an aqueous salt solution. This salt solution plus residual monomer and other trace organics must be removed before the resin can be used for sampling trace organics.

Transfer the resin to a large Soxhlet extractor with a 1.5-liter dumping volume. This requires 2 to 2.5 liters of solvent in the 3-liter supply flask. The XAD-2 resin is then extracted in sequence with the following solvents and times:

- Water - 22 hrs
- Methanol - 22 hrs
- Anhydrous ether - 8 hrs
- Pentane - 22 hrs

The water removes the salt solution and any water soluble organic material. Methanol removes the residual water from the resin and ether removes the majority of the polar organic material. Pentane is used as the final stage because it is the solvent used in the actual extraction of collected material from the resin.

After the final pentane extraction, transfer the XAD-2 resin into a clean flask and dry it under a vacuum for 18 hours using mild heat from a heat lamp.

APPENDIX C

MFG. ORGANIC/MAJOR MASS PEAKS

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Aliphatic Hydrocarbons			
A. Alkanes and Cyclic Alkanes			
Methane		16	16(100), 15(86), 14(16), 13(8)
Ethane		30	28(100), 27(33), 30(26), 26(23)
Propane		44	29(100), 28(62), 44(40), 43(34)
Butanes	N-Butane	58	43(100), 29(41), 27(31), 28(30)
Pentanes	N-Pentane	72	43(100), 42(60), 41(40), 27(35)
Cyclopentane		70	42(100), 70(30), 41(29), 55(29)
Hexanes	N-Hexane	86	57(100), 43(82), 41(74), 29(63)
Cyclohexane		84	56(100), 84(73), 41(57), 55(34)
Heptanes	N-Heptane	100	43(100), 41(52), 57(48), 29(46)
Octanes	N-Octane	114	43(100), 41(33), 29(34), 57(34)
Nonanes	N-Nonane	128	43(100), 57(67), 41(40), 29(37)
Alkanes (C>9)	N-Decane	142	43(100), 57(82), 41(43), 29(38)
B. Alkenes, Cyclic Alkenes and Dienes			
Ethylene		28	28(100), 27(54), 26(50), 25(7)
Propylene		42	41(100), 42(69), 39(61), 27(25)
Butylenes	Cis 2-Butene	56	41(100), 56(48), 39(36), 27(33)
Butadienes	1,3 Butadiene	54	54(100), 39(91), 53(66), 27(46)
Pentenes	1-Pentene	70	42(100), 55(58), 41(45), 39(35)

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Cyclopentadienes	1,3 Cyclopentadiene	66	66(100), 65(65), 39(50), 40(41)
Hexenes	1-Hexene	84	41(100), 56(86), 42(75), 27(68)
Cyclohexene		82	67(100), 54(77), 82(40), 41(37)
Cyclohexadiene	1,3 Cyclohexadiene	80	79(100), 80(57), 77(38), 39(23)
Heptenes	1-Heptene	98	41(100), 56(87), 29(71), 55(60)
C. Alkynes			
Acetylene		26	26(100), 25(20), 13(6), 24(6)
Propyne		40	40(100), 39(92), 38(36), 37(28)
Butynes	2-Butyne	54	54(100), 27(45), 53(45), 39(26)

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Alkyl Halides			
A. Saturated Alkyl Halides			
Methyl bromide		94	94(100), 15(47), 93(21), 91(7)
Methyl chloride		50	50(100), 15(83), 52(32), 49(10)
Methyl iodide		142	142(100), 127(38), 141(14), 15(13)
Dichloromethane (methylene chloride)		84	49(100), 84(58), 86(36), 51(30)
Bromodichloremethane		162	83(100), 85(64), 47(23), 48(16)
Dibromochloromethane		206	129(100), 127(78), 131(25), 208(14)
Tribromomethane (bromoform)		250	173(100), 171(50), 175(49) 93(22)
Dibromodichloromethane		240	163(100), 161(62), 165(45), 79(22)
Dichlorodifluoromethane		120	85(100), 87(33), 50(12), 101(9)
Trichlorofluoromethane		136	101(100), 103(60), 35(16), 66(15)
Carbon tetrachloride		152	117(100), 119(97), 121(32), 82(19)
1,2-dichloroethane (ethylene chloride)		98	62(100), 27(93), 49(37), 64(32)
1,1,1,-trichloroethane		132	97(100), 99(64), 61(50), 117(19)

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
1,2-dichloro-1,2-difluoro	$C_2Cl_2F_2$	134	
Hexachloroethane		234	117(100), 119(96), 201(80), 203(51)
Dichloropropanes	1,2-Dichloropropanes	112	63(100), 62(71), 27(57), 41(48)
Bromobutanes	1-Bromobutane	136	57(100), 41(49), 29(34), 27(20)
Hexachlorocyclohexane (lindane)		288	181(100), 183(83), 217(66), 219(65)
1-chlorooctane		148	91(100), 43(54), 55(39), 41(39)
B. Unsaturated Alkyl Halides			
Chloroethene (vinyl chloride)		62	62(100), 27(67), 64(31), 26(19)
1,2-dichloroethene		96	61(100), 96(73), 98(47), 63(32)
1,1-dichloroethene (vinylidine chloride)		96	61(100), 96(61), 98(32), 63(32)
Tetrachloroethene (perchloroethylene)		164	166(100), 164(79), 129(69), 131(66)
Dichloropropenes	2,3 Dichloropene	110	75(100), 39(67), 71(32), 110(23)
1,3-Hexachlorobutadiene		258	225(100), 227(65), 223(63), 190(42)
Hexachlorocyclopentadiene		270	237(100), 235(64), 239(63), 95(42)

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Ethers			
2 2'-Oxy bis propane (Diisopropylether)		102	45(100), 43(39), 47(24), 59(11)
2-Ethyl-4-methyl-1,3-Dioxolane		130	
1,3-Dioxane		88	28(100), 31(81), 29(79), 87(58)
1,4-Dioxane		88	28(100), 29(37), 88(31), 58(24)
2-Methoxy biphenyl (O-phenylanisole)		184	184(100), 169(46), 141(24), 115(10)

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Halogenated Ethers			
Chloromethyl methyl ether		80	45(100), 29(43), 15(39), 49(14)
1,1'-Dichloromethyl ether		114	79(100), 49(47), 81(33), 51(16)
2-Chloro-1,3-epoxypropane (Bepichlorohydrin)		92	
2-Chloroethylmethyl ether		94	
1-Chloro-1,3-oxetane		92	
Chloromethylethyl ether		94	
Chloro ethyl ethyl ether		108	
1.1'-Dichlorodiethyl ether		142	
1.2-Dichlorodiethyl ether		142	
1,2-Dichloroethyl ethyl		142	
1,2-Dichloroethyl ethyl ether			
2,2'-Dichlorodiethyl ether		142	93(100), 63(98), 27(75), 95(32)
α-Chlorobutylethyl ether		136	
bis-(1-chloroisopropyl) ether		170	
1,2-Dichlorodiisobutyl ether		-	
Bromophenyl phenyl ether		248	

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Alcohols			
A. Primary Alcohols			
Methyl alcohol		32	31(100), 32(72), 29(42), 28(9)
Ethyl alcohol		46	31(100), 45(35), 29(27), 27(24)
1-propanol		60	31(100), 27(19), 29(18), 59(12)
1-butanol		74	31(100), 56(90), 41(74), 43(64)
1-pentanol		88	42(100), 55(74), 41(60), 70(56)
B. Secondary Alcohols			
2-propanol		60	45(100), 43(19), 27(17), 29(12)
2-butanol		74	45(100), 27(22), 31(22), 59(20)
2-pentanol; 3-pentanol	2 pentanol	88	45(100), 43(16), 55(16), 27(13)
α-hydroxytoluene (m-cresol)		108	108(100), 107(94), 79(35) 39(32)
2,6-dimethyl-4-heptanol		144	69(100), 87(49), 45(43), 43(42)
Borneol		154	95(100), 41(30), 27(20), 43(18)
C. Tertiary Alcohols			
2-methyl propanol (tert-butyl alcohol)		74	59(100), 31(28), 41(18), 43(12)
α-methyl- hydroxytoluene		122	122(100), 107(83), 121(38), 77(32)

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
α-terpineol		154	59(100), 43(74), 93(72), 121(52)
Isoborneol		154	95(100), 41(42), 27(25), 43(24)

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Glycols, Epoxides			
A. Glycols			
Ethylene glycol (1.2-Ethanediol)		62	31(100), 33(32), 29(14) 32(10)
Propylene glycol (1.2-Propanediol)		76	45(100), 43(14), 31(12), 27(9)
B. Epoxides			
2,3-epoxy-1 propanol (glycidol)		74	44(100), 43(89), 31(59), 18(44)
1-chloro-2,3-epoxy propane (α-Epichlorohydrin)		92	57(100), 27(39), 29(31), 49(25)

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Aldehydes, Ketones			
A. Aldehydes			
Formaldehyde		30	29(100), 30(88), 28(31), 14(4)
Acetaldehyde		44	29(100), 44(88), 43(50), 42(15)
Propionaldehyde		58	29(100), 28(69), 27(58), 58(27)
Acrolein		56	27(100), 56(65), 26(59), 28(53)
Butyraldehyde		72	44(100), 43(79), 72(73), 41(60)
Bromoform butanol		?	Improper Name?
3-Methylbutanol		88	41(100), 29(98), 57(83), 31(72)
Benzaldehyde		106	77(100), 106(91), 105(89), 51(58)
B. Ketones			
Acetone		58	43(100),58(37), 42(7), 27(5)
Tetrachloroacetone		194	83(100), 85(65), 111(21), 113(14)
Butanone		72	43(100), 72(21), 29(17), 27(9)
Isophrone (Isophorone)		138	82(100), 39(88), 138(17), 41(13)
Camphor (d)		152	126(100), 95(97), 41(79), 81(71)
Acetophenone		120	105(100), 77(83), 51(30), 120(25)

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Chlorohydroxy benzophenone		232	155(100), 232(58), 105(57), 157(32)
5,6-Benzo-9-anthrone		?	Unknown
Dihydro(d)carvone		152	81(100), 67(70), 41(59), 39(46)

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Carboxylic Acids and Derivatives			
A. Carboxylic Acids			
Formic acid		46	29(100), 46(61), 45(48), 28(17)
Acetic acid		60	43(100), 60(99), 45(98), 44(34)
Maleic acid		116	18(100), 26(99), 54(77), 25(24)
Benzoic acid		122	105(100), 122(78), 77(75), 51(46)
Phthalic acid		166	149(100), 121(25), 166(18), 105(16)
Long chain acids	C_{12}	200	73(100), 60(93), 41(42), 43(59)
B. Carboxylic Acids with Additional Functional Groups			
Hydroxyacetic acid (glylolic acid)		76	18(100), 31(79), 32(24), 17(22)
Hydroxybenzoic acids (p)		138	28(100), 121(26), 37(14), 138(21)
3-hydroxypropanoic acid, (β-lactone)		72	47(100), 28(93), 26(28), 43(78)
C. Amides			
Formamide		45	18(100), 45(56), 28(44), 29(30)
Acetamide		59	44(100), 59(93), 43(75), 15(48)

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
6-aminohexanoic acid (6-aminocaproic acid)		131	44(100), 59(93), 43(75), 15(48)
D. Esters			
Methyl methacrylate		100	41(100), 69(68), 39(38), 100(31)
Phthalates (Diethyl phthalate)		222	149(100), 177(28), 150(13), 176(9)
Adipates (Diethyl adipate)		202	29(100), 55(57), 111(52), 27(50)
Long chain esters		-	Too vague -
Methyl benzoate		136	105(100), 71(68), 51(38), 136(28)
Phenyl benzoate		198	105(100), 77(29), 106(8), 51(7)
Di-2-ethylhexyl phthalate (Dop)		392	149(100), 167(41), 57(40), 43(34)
6-Aminohexanoic acid (e-caprolactam)		113	

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Nitriles			
Acetonitrile		41	41(100), 40(54), 39(18), 15(1)
1-Cyanoethane (acrylonitrile)		53	26(100), 53(99), 52(75), 51(32)
1,4-Dicyano-1-hydroxy butane		124	
Benzonitrile		103	103(100), 76(32), 50(17), 51(10)
Naphthonitriles		153	

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Azo Compounds, Hydrazine, and Derivatives			
Diazomethane		42	
Hydrazine		32	32(100), 31(44), 29(40), 30(31)
Dimethyl hydrazines		60	60(100), 42(98), 28(52), 45(52)
Diphenylhydrazines		184	169(100), 168(56), 167(32) 51(26)

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Amines			
A. Primary Amines			
Ethylamine		45	30(100), 28(29), 44(20), 45(19)
Ethanolamine		61	42(100), 61(78), 43(49), 44(29)
3-Aminopropene		57	30(100), 56(80), 28(76), 57(32)
Propanolamine (iso)		75	44(100), 42(14), 58(5), 41(5)
1-Aminobutane		73	30(100), 28(9), 41(6), 27(5)
2-Aminobutane		73	44(100), 18(15), 58(11), 41(11)
Cyclohexylamine		99	56(100), 43(30), 28(15), 30(14)
1,2-Diaminoethane		60	30(100), 18(13), 42(6), 43(5)
Aniline		93	93(100), 66(33), 65(18), 39(18)
2-Aminotoluene		107	28(100), 106(84), 107(66), 77(24)
Dimethylaniline		121	120(100), 121(70), 77(25), 51(16)
Anisidines (p-)		123	108(100), 123(68), 80(41), 53(22)
Aminodiphenyl		169	168(100), 167(56), 51(53), 169(52)
Aminonaphthalenes (2-)		143	143(100), 115(36), 144(13), 116(13)

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
1,4-Diaminobenzene		108	108(100), 80(82), 52(39), 28(38)
4,4'-Diaminodiphenyl		184	
3,3'-Dichlorobenzidine		252	252(100), 254(67), 126(16), 77(15)
4,4'-Methylene bis (2-chloroaniline)		?	?
B. Secondary Amines			
Ethyleneimine		43	42(100), 28(79), 43(55), 15(36)
Dimethylamine		45	44(100), 28(68), 45(51), 15(20)
Ethylmethylamine		59	
Diethylamine		73	58(100), 30(98), 28(37), 27(29)
Morpholine		87	57(100), 29(99), 87(69), 28(69)
		121	120(100), 121(70), 77(25), 51(16)
C. Tertiary Amines			
N,N-Dimethylaniline		121	120(100), 121(68), 77(25), 105(13)

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Nitrosamines			
Dimethylamine-N-Nitroso		74	42(100), 74(88), 43(52), 44(21)
Diethylamine-N-Nitroso		102	42(100), 44(98), 102(74), 57(48)
Dipropylamine-N-Nitroso		130	43(100), 42(66), 70(66), 41(47)
Diisopropylamine-N-Nitroso		130	43(100), 70(33), 42(32), 41(31)
Dipentylamine-N-Nitroso		158	
Aniline, N-Methyl-N-Nitroso		136	136(100), 107(56), 77(54), 105(38)
Diphenylamine-N-Nitroso		198	169(100), 168(72), 167(50), 51(19)

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Mercaptans, Sulfides and Disulfides			
A. Mercaptans			
Methyl mercaptan		43	47(100), 48(90), 45(47), 46(12)
Ethyl mercaptan		62	62(100), 29(90), 47(80), 27(80)
Propyl mercaptans		76	47(100), 76(88), 43(80), 42(74)
Butyl mercaptans		90	56(100), 41(92), 27(57), 90(51)
Benzenethiol		110	110(100), 66(38), 109(25), 51(22)
1-anthrathiol		210	
Perchloromethyl mercaptan		150	
B. Sulfides, Disulfides			
Dimethyl sulfide		62	47(100), 62(83), 45(59), 46(34)
Diethyl sulfide		90	75(100), 47(82), 20(72), 29(63)
DiPhenyl sulfide		186	186(100), 185(46), 51(26), 184(23)
DiMethyl disulfide		94	94(100), 45(63), 79(59), 46(38)

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Sulfonic Acids, Sulfoxides			
A. Sulfonic Acids			
Benzenesulfonic acid		158	158(100), 77(85), 94(74), 51(39)
9,10-Anthraquinone-disulfonic acids		368	
B. Sulfoxides			
Dimethyl sulfoxide		78	63(100), 78(68), 15(40), 45(35)

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Benzene, Substituted Benzene Hydrocarbons			
Benzene		78	78(100), 52(17), 51(16), 77(15)
Toluene		92	91(100), 92(76), 39(19), 65(13)
Ethyl benzene		106	91(100), 106(31), 51(13), 39(10)
Styrene		104	104(100), 103(39), 98(32), 51(30)
N-Propyl benzene		120	91(100), 120(24), 92(11), 65(9)
Isopropyl benzene (cumene)		120	105(100), 120(26), 77(88), 51(10)
N-Butyl benzene		134	91(100), 92(55), 134(24), 27(12)
Biphenyl		154	154(100), 153(32), 152(24), 76(18)
4,4'-diphenylbiphenyl (P-P, quaterphenyl)		306	306(100), 307(26), 153(26), 152(6)
Xylenes		106	91(100), 106(62), 105(30), 77(12)
Dialkyl benzenes		-	Vague description
Tetrahydronaphthalene (Tetralin)		132	104(100), 132(64), 91(50), 13(17)

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Dihydronaphthalene		130	128(100), 130(95), 129(78), 115(70)
Terphenyl		230	230(100), 231(19), 115(15), 228(12)
Trimethyl benzene		120	105(100), 120(59), 119(16), 39(14)
Tetramethyl benzene		134	119(100), 134(58), 43(23), 42(17)

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Halogenated Aromatic Compounds			
A. Ring Substituted Aromatics			
Chlorobenzene		112	112(100), 77(48), 114(32), 51(16)
dibromobenzenes		234	236(100), 234(51), 238(49), 155(30)
Bromochlorobenzenes		190	192(100), 190(76), 111(57), 75(28)
1,2-dichlorobenzene		146	146(100), 148(64), 111(36), 75(20)
1,3-dichlorobenzene		146	146(100), 148(65), 111(32), 75(17)
1,4-dichlorobenzene		146	146(100), 148(65), 111(32), 75(19)
Polychlorinated benzenes		-	Strong molecular ion - loss of Cl
Chloronaphthalene		162	162(100), 126(13), 77(10), 63(10)
Polychlorinated biphenyls		-	Strong molecular ion - loss of Cl
Bromo benzene		156	77(100), 156(77), 158(76), 51(42)

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
B. Aromatics with Halogenated Alkyl Side Chain			
α-chlorotoluene (benzyl chloride)		126	91(100), 126(28), 65(9), 63(6)
bis-(chloromethyl)-benzene		174	139(100), 141(32), 174(21), 103(18)

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Aromatic Nitro Compounds			
Nitrobenzene		123	77(100), 51(59), 123(42), 50(25)
4-nitrobiphenyl; nitrobiphenyls		199	
Chloronitrobenzenes		157	111(100), 157(73), 75(52), 113(32)
Methoxynitrobenzenes		153	
Dinitrotoluenes		182	

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Phenols			
A. Monohydrics			
Phenol		94	94(100), 66(28), 39(28), 65(22)
Methyl phenols (cresols) (p-cresol)		108	107(100), 108(75), 77(30), 79(26)
2-methoxyl phenol		124	124(100), 94(57), 81(39), 39(31)
Ethylphenols (O-ethylphenol)		122	107(100), 122(40), 77(18), 79(9)
Hydroxybiphenyls		170	170(100), 169(60), 141(24), 115(15)
Dimethylphenols (Xylenols) (2,5)		122	122(100), 107(87), 121(43), 77(30)
Polyalkylphenols		-	Too vague
B. Dihydrics, Polyhydrics			
2,2'-dihydroxydiphenyls		186	186(100), 157(28), 158(23), 131(22)
1,2-dihydroxybenzene (catechyl)		110	110(100), 64(39), 63(22), 52(15)
1,3-dihydroxybenzene (resorcinol)		110	110(100), 81(22), 39(22), 53(19)
1,4-dihydroxybenzene (hydroquinone)		110	110(100), 53(27), 81(22), 55(22)

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
1,2,3-trihydroxybenzenes		126	126(100), 108(33), 80(30), 52(22)
C. Fused ring hydroxy compounds			
α-naphthanol		144	144(100), 115(74), 116(37), 145(10)
β-naphthanol		144	144(100), 115(63), 116(28), 57(15)
Phenthrols (Phenanthrols)		194	43(100), 194(94), 165(78), 39(22)
Indanols		134	
Acenaphthenols		170	
2-hydroxyfluorene		182	
2-hydroxydibenzofuran		184	

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Halophenols			
2-chlorophenol		128	128(100), 64(35), 130(32), 63(14)
2,4-dichlorophenol		162	162(100), 164(63), 63(30), 98(27)
Pentachlorophenol		264	
Chlorinated cresols		144	

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Nitrophenols			
2-nitrophenol		139	139(100), 65(36), 64(22), 63(22)
3-nitrophenol		139	65(100), 39(88), 139(74), 93(70)
4-nitrophenol		139	139(100), 65(35), 64(20), 63(21)
Dinitrophenols		184	
o-cresol, dinitro		198	
p-cresol, dinitro		198	198(100), 182(36), 77(35), 51(27)
2-amino-4,6-dinitrophenol		199	
Trinitrophenol		229	

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Fused Polycyclic Hydrocarbons			
Naphthalene		128	128(100), 51(12), 129(11), 64(11)
Monoalkyl naphthalenes		-	Too vague
Phenyl naphthalenes (α)		204	204(100), 203(68), 202(47), 101(34)
Dimethyl naphthalenes		156	156(100), 141(26), 155(30), 153(14)
Acenaphthene; acenaphthylene		154	154(100), 153(95), 152(53), 76(26)
Anthracene		178	178(100), 176(16), 179(16),
2,7-dimethylanthracene		206	
Phenanthrene		178	178(100), 179(15), 176(15), 89(14)
Methylphenanthrenes		192	
Naphthacene		228	228(100), 229(30), 114(29), 226(21)
1,2-benzanthracene		228	228(100), 229(19), 226(19), 114(18)
9,10-dimethyl-1,2-benzanthracene		256	256(100), 241(40), 239(37), 240(24)
Benzo(c)phenanthrene		228	228(100), 226(45), 227(34), 118(31)

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Chrysene		228	228(100), 226(22), 229(19), 114(16)
Methylchrysenes		242	242(100), 239(20), 243(20), 241(17)
Triphenylene (9,10 benzphenanthracene		228	228(100), 226(22), 229(21), 113(17)
Pyrene		202	202(100), 101(26), 208(17), 100(17)
1-methyl pyrene		216	216(100), 215(61), 94(26), 217(18)
1,2-benzonaphthacene		278	
Benzo(b)chrysene		278	278(100), 276(81), 138(25), 279(22)
1,2:3,4-dibenzanthracene		278	278(100), 139(24), 279(24), 276(15)
1,2:5,6-dibenzanthracene		278	278(100), 139(24), 279(24), 276(16)
Benzo[a]pyrene		252	252(100), 126(23), 253(21), 250(16)
Benzo[e]pyrene		252	252(100), 126(23), 252(21), 250(16)
Perylene		252	252(100), 253(22), 126(21), 250(21)
Picene (dibenzo(a,i)phenanthrene)		278	

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Dibenzo[a,h]pyrene		302	
Dibenzo[a,i]pyrene		302	
Dibenzo[a,ℓ]pyrene		302	
Benzo[g,h,i]perylene		276	276(100), 138(37), 277(55)
Coronene		300	300(100), 150(78), 149(66), 148(37)

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Fused Non-Alternant Polycyclic Hydrocarbons			
Idene and derivatives (alkyl)	Indene	116	116(100), 115(84), 63(14), 39(11)
Dicyclopentadiene		130	
Fluorene and derivatives (alkyl)	Fluorene	166	166(100), 165(81), 163(15), 164(14)
Cyclopentanonaphthalene			
2,3-benzofluorene		216	216(100), 215(78), 217(18), 107(16)
Fluoranthene		202	202(100), 101(22), 203(17), 200(14)
1,2-benzofluorene		216	216(100), 215(62), 107(21), 217(19)
4-H-cyclopenta(def)phenanthrene			
Benzo(k)fluoranthene		252	252(100), 253(23), 125(16),
Benzo(e)fluoranthene		252	Similar to (k)
Benzo(j)fluoranthene		252	Similar to (k)
1,2:5,6-dibenzofluorene		266	
3-methyl cholanthrene		268	268(100), 252(39), 253(39), 267(24)
Indeno (1,2,3,c,d)pyrene		276	276(100), 138(28), 277(27)

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Truxene (tribenzylene benzene)		?	
Tetrahydrofluoranthene		206	178(100), 206(67), 89(28), 76(19)

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Heterocyclic Nitrogen Compounds			
A. Pyridine and Substituted Pyridines			
Pyridine		79	79(100), 52(74), 51(36), 50(26)
Monosubstituted alkyl pyridines		93	93(100), 66(41), 39(31), 92(20)
phenyl pyridines		155	
Chloropyridine		113	113(100), 78(49), 115(32), 18(24)
Disubstituted, polysubstituted pyridines (dimethyl pyridine)		107	107(100), 106(65), 39(32), 79(29)
B. Fused 6-membered Ring Heterocycles			
Quinoline, isoquinoline		129	129(100), 102(22), 51(18), 128(16)
Methylquinolines, methylisoquinolines		143	143(100), 142(43), 28(24), 115(16)
Dimethylquinolines, dimethylisoquinolines		157	157(100), 156(33), 158(13), 115(10)
Acridine		179	179(100), 178(14), 180(14), 89(12)
Dihydroacridine		181	

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Benzo(c)quinoline		179	179(100), 178(24), 180(16), 151(12)
Benzo(f)quinoline		179	179(100), 178(24), 151(17), 76(16)
Benzo(h)quinoline		179	179(100), 178(24), 180(14), 151(11)
Benz(a)acridine		229	
Benz(c)acridine		229	
Dibenz(a,j)acridine		279	
Dibenz(a,h)acridine		279	
Dibenz(c,h)acridine		279	
Indeno(1,2-b)quinoline			
Indeno(1,2,3,i,j)isoquinoline			
C. Pyrrole and Fused Ring Derivatives of Pyrrole			
Pyrrole		67	67(100), 41(58), 39(58), 40(51)
Indole		117	117(100), 90(40), 89(24), 63(14)
Methylindoles		131	130(100), 131(59), 77(14), 65(10)
Carbazole		167	167(100), 166(18), 83(14), 168(13)

Table C-1. MEG Organic/Major Mass Peak

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Methylcarbazoles (9-)		181	
Benzo(a)carbozole		231	
Dibenzo(a,g)carbozole		281	
Dibenzo(a,i)carbozole		281	
Dibenzo(c,g)carbozole		281	
D. Nitrogen Heterocycles containing additional Heteroatoms			
Benzothiazole		135	135(100), 108(35), 69(27), 63(13)
Methyl benzothiazoles		149	148(100), 108(32), 69(29), 149(17)

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Heterocyclic Oxygen Compounds			
Furan		68	39(100), 68(71), 37(18), 29(16)
Benzofurans		118	118(100), 90(30), 89(29), 63(13)
Dibenzofuran		168	168(100), 139(23), 169(13), 84(11)
Methyldibenzofurans		182	
Naphthofurans		168	
Benzo[b]naptho[2,3-d]furan		218	
Phenanthro[9,10-b]furan		218	
1,9-benzoxanthene		232	

Table C-1. MEG Organic/Major Mass Peaks

Category	Compound Detail	Molecular Weight	M/e Values (RI)
Heterocyclic Sulfur Compounds			
Thiophene		84	84(100), 58(65), 45(55), 39(28)
Methylthiophenes		98	97(100), 98(55), 45(21), 39(17)
Dimethylthiophenes		112	111(100), 112(78), 97(58), 59(22)
Trimethyl Thiophene		126	111(100), 126(12), 125(58), 45(27)
2,2'-Bithiophene		166	166(100), 121(29), 45(26), 69(16)
Benzo[b] thiophene		134	134(100), 89(10), 135(10), 63(8)
Dibenzothiophene		184	184(100), 185(14), 139(12), 92(11)
Benzonaphthathiphene		234	234(100), 235(18), 117(18), 232(9)
Tetramethylthiophenes		140	

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